DYERS' MATERIALS

P. HEERMANN

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DYERS' MATERIALS

AN INTRODUCTION TO THE EXAMINATION EVALUATION AND APPLICATION OF THE MOST IMPORTANT SUBSTANCES USED IN DYEING, PRINTING, BLEACHING AND FINISHING

BY

PAUL HEERMANN, Ph.D.

TRANSLATED BY

ARTHUR C. WRIGHT, M.A. (Oxon.), B.Sc. (Lond.)
FORMERLY ASSISTANT LECTURER AND DEMONSTRATOR IN CHEMISTRY AT THE YORKSHIRE COLLEGE, LEEDS
AUTHOR OF "SIMPLE METHODS FOR TESTING PAINTERS' MATERIALS," ETC.

WITH TWO PLATES

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PREFACE.

The present work arises from the author's long connection with the chemical textile industries and from the want which he has found of a special text-book on the examination and evaluation of the materials used in those industries, which should contain, within a brief compass, the most valuable methods and data for the use of dyers' and technical chemists.

The need for such a book appears to be evident, since the ordinary works on technical analysis cannot deal adequately with a special branch, which has a constantly increasing interest for the chemist, whilst, on the other hand, most text-books on dyeing must give the principal attention to the tinctorial side of the matter.

The book is intended in the first place for the chemist, since it presupposes a knowledge of the principles of chemical analysis, frequently only indicates common methods, and bestows chief attention on the specific articles and methods of examination. It should, also, however, be useful for educational purposes, and should be of service, by the side of general analytical text-books, to students in technical schools and colleges.

The colouring processes of dyeing have been excluded from this book, since they form a separate

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branch, and thus should be treated in a special volume, but also because there does not exist a particular want of such a book, in view of the flourishing literature of that branch.

On the other hand, the author has deemed it advisable to preface the special part of the book by a general part dealing—generally in tabular form—with indicators and solutions, and by a short description of the prime necessaries of the textile industries—water and the textile fibres. The chapter, usual in detailed text-books, on preparation for analysis, sampling, etc., has been omitted.

Whilst the author publishes the book in the hope that it will meet with good acceptance, he will be thankful for hints in regard to possible additions and alterations. Also in this place he records his best thanks to certain gentlemen who have assisted him by private communications; in particular, he expresses his warmest thanks to his revered former teacher, Dr. H. Lange, Director of the Crefeld Higher Dyeing School.

THE AUTHOR.

CREFELD, GERMANY

TRANSLATOR'S PREFACE.

THE only changes from the original, which have been made in the translation, consist in the insertion of references to English sources of information and the substitution of the most recent table of atomic weights. These references are, as a rule, given in addition to the original German source; this is the case with original papers abstracts of which have appeared in the *Journal of the Society of Chemical Industry (J.S.C.I.).* References to Fresenius, *Quant. Anal.*, are to the English edition.

A. C. WRIGHT.

HULL, *September*, 1901.

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CHAPTER I.

GENERAL.

Indicators.—The correct choice of an indicator from among the large number known is of great importance if results are to be reliable and accurate. The indispensable indicators are strictly limited in number; the choice is often a personal matter: an indicator, which would be absolutely rejected by one chemist, would be preferred by another. This is due to differences in eyesight.

A random use of indicators in technical analysis is most reprehensible; in order to obtain uniform results it is necessary to use the customary solutions of indicators and not to depart from them without cogent reasons and previous comparative analyses. Otherwise considerable differences may occur (phenolphthaleïn and rosolic acid, phenolphthaleïn and methyl orange). Further, if the reaction is to show a sharp end-point, the indicator must be absolutely pure, or at least as pure as it can be obtained. This is especially the case with litmus, turmeric, indigo solution, etc.; *e.g.*, Neubauer and von Schröder, and J. König (*Zeits. für angew. Chem.*, 1891, 108) found that certain commercial preparations of indigo carmine were quite useless for titrations with potassium permanganate, since in the end-reaction they did not change sharply from green to yellow, but to uncertain brownish-red shades. The best test of purity is afforded by a practical application.

For purposes of rapid comparison and review, the properties, solutions and reactions of the best-known indicators are given in tabular form (see Table I., facing p. 4).

Standard Solutions.

The standard solutions, which are in daily use, may either be normal (twice, semi- or deci-normal) or may just as well be simply standardised solutions of any definite strength, *e.g.*, 1·125 normal, 0·978 normal, etc. In the latter case the number of c.c. of solution used must be multiplied by the proper factor, in order to obtain the corresponding number of c.c. of normal solution. The one or the other method is preferred according to individual circumstances.

In the preparation of normal or standardised solutions some initial standard substance is required, by means of which the strength of the solution is checked; for this purpose the following substances are used in acidimetry:—

1. Crystallised oxalic acid (to be used with caution, since it may be partially effloresced or contain excess of water, which conditions are not readily recognised).
2. Anhydrous oxalic acid.
3. Fused sodium carbonate (obtained by igniting sodium bicarbonate).
4. Marble.
5. Potassium bitartrate.
6. Sodium chloride.

All these standard substances must be qualitatively tested for purity; in general those which can be ignited—sodium chloride, carbonate, etc.—are to be preferred, since they cannot contain any unknown quantity of water. When the chemically pure compound has been dissolved, the solution is titrated by it in the known manner, using a sensitive indicator; the opposite solutions are then standardised.

This is the case with oxalic acid, soda and potassium tartrate. With sodium chloride a normal or deci-normal solution is made, by means of which solutions of silver nitrate and ammonium sulphocyanide are standardised, from which a hydrochloric acid solution is standardised by Volhard's method (hydrochloric acid + excess of silver nitrate, titrated back by ammonium sulphocyanide, using iron alum as indicator).

From each of the acids alkaline solutions may be standardised and thence other acids, diluted if desired, etc.

REAGENTS.

Table II., giving the composition and applications of solutions and reagents in ordinary use, immediately follows Table I. (see p. 4).

APPENDIX.

In an Appendix are given Tables of the atomic weights of the elements, the molecular weights of important compounds and certain gravimetric and volumetric equivalents.

A Normal[1] Solution contains Grammes per Litre.		Generally used as	Original Standard Substance.	Titrated against	Indicator.
Sulphuric acid	49·0	$\frac{2}{1}$, $\frac{1}{1}$, $\frac{1}{2}$, $\frac{1}{10}$ N	Oxalic acid, soda, marble, potassium bitartrate	Caustic soda	Litmus, phenolphthaleïn, etc.
Hydrochloric acid	36·4	$\frac{1}{1}$, $\frac{1}{10}$ N	Do., also sodium chloride	Do.	Do.
Nitric acid	63·0	$\frac{1}{1}$ N.	Do.	Do.	Do.
Oxalic acid	63·0	$\frac{1}{1}$, $\frac{1}{5}$, $\frac{1}{10}$, $\frac{1}{100}$ N	Oxalic acid	Do.	Phenolphthaleïn
Caustic soda	40·0	$\frac{2}{1}$, $\frac{1}{1}$, $\frac{1}{2}$, $\frac{1}{10}$ N	Oxalic acid, etc., as above	Sulphuric acid, oxalic acid	Litmus, etc.
Sodium carbonate	53·0	$\frac{1}{1}$, $\frac{1}{3}$ N	Sodium carbonate, or as above	Do.	Methyl orange
Ammonia	17·0	$\frac{1}{2}$, $\frac{1}{10}$ N	Oxalic acid, soda, etc.	Do.	Litmus, lacmoid, turmeric
Silver nitrate	170·0	$\frac{1}{10}$ N	Sodium chloride	Sodium chloride	Neutral potassium chromate
Sodium chloride	58·5	$\frac{1}{10}$ N	Do.	Silver nitrate	Do.
Potassium permanganate	31·6	$\frac{1}{5}$, $\frac{1}{25}$, $\frac{1}{100}$ N	Oxalic acid, iron wire	Oxalic acid, ferrous salt	Is its own indicator
Sodium thiosulphate	248	$\frac{1}{10}$ N	Iodine	Iodine solution	Starch solution
Iodine	127	$\frac{1}{10}$, $\frac{1}{25}$, $\frac{1}{100}$ N	Do.	Thiosulphate	Do.
Arsenious acid (sodium arsenite)	49·5	$\frac{1}{10}$ N	Do.	Iodine solution	Do.
Potassium bichromate	49·13	$\frac{1}{5}$, $\frac{1}{10}$ N	Ferrous salt, ferrous ammonium sulphate	Ferrous salt	Potassium ferricyanide
Indigo solution		10 c.c. should correspond to 0·001 gramme N_2O_5. See estimation of N_2O_5 under *Water*	Potassium nitrate	Potassium nitrate solution	Is its own indicator

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[1] In the table, and afterwards, N is used as a contraction of normal; thus 2 N sulphuric acid, N sulphuric acid and $\frac{1}{10}$ N sulphuric acid represent twice normal, normal and decinormal sulphuric acid respectively.

TABLE I.—*Indicators.*

Indicator.	Solvent.	Strength of Solution.	Used for 100 c.c. of Liquid.	Temperature.	Suitable for the Titration of	Less Suitable for	Unsuitable for the Titration of	The Influence Predominates of	Required to give a Distinct Reaction.	Reactions and Remarks.	Literature.
			c.c.								
Litmus	Water	1 : 10	0·2	Cold and hot. With nitrous, lactic and tartaric acids only *cold*	Sulphuric, hydrochloric, nitric, sulphurous, nitrous, oxalic, lactic and tartaric acids, acids in the presence of organic bases (aniline, toluidine, quinoline), etc.	Alkalis in the presence of alumina, soda, alkaline silicates, alkaline sulphides	Sulphites, borates, carbonic, phosphoric, arsenic, silicic, arsenious and chromic acids, organic bases and acids, alkaline solutions of alumina, sodium aluminate, aniline, toluidine; in the presence of sulphates and chlorides of the heavy metals	Acids	0·5 c.c. of $\frac{N}{100}$ HCl, 1·0 c.c. of $\frac{N}{100}$ KOH	Anhydrides and anhydrous acids without action; $NaNO_2$ is neutral; K_2CrO_4 slightly alkaline; Na_2HPO_4 and Na_2HAsO_4 alkaline; NaH_2PO_4 acid; alkaline sulphites allow only 17 per cent. to be found. Sulphates and chlorides of the heavy metals react acid	Mohr and Classen, *Titrir-methoden*. Wartha, *Ber.*, **9**, 217, and *Zeits. anal. Chem.*, 1876, 322. Förster, *Zeits. anal. Chem.*, 1889. 428. Kretschmar, *Zeits. anal. Chem.*, 1880, 341. Stutzer, *Böckmann I.*, 116. Dubois, *Bull. Soc. Chim.*, **49**, 963. Lunge, *Dingl. Polyt. Journ.*, **251**, 40. Plugge, *Arch. Pharm.*, 1887, 45 and 49. Marsh, *Chem. News*, **61**, 2. D. R. Brown, *Pharm. Journ. and Trans.*, 1896, 56, 181
Phenolphthalein	Alcohol	1 : 100	0·5	Cold (with H_2SO_4, HCl, HNO_3, SO_2, CrO_3). Oxalic acid also hot	Sulphuric, hydrochloric, nitric, sulphurous, phosphoric, nitrous, chromic, arsenic and organic acids, alkalies in the presence of alumina, caustic alkalis, alkaline sulphides, etc.	Carbonic acid in dilute solutions, acids united to organic bases	Arsenious, silicic and boric acids, organic bases, ammonia, aniline, toluidine; in strong salt solutions	Acids	0·5 c.c. $\frac{N}{100}$ NaOH	$NaHCO_3$, K_2CrO_4, Na_2SO_3, $NaC_2H_3O_2$, Na_2HPO_4, Na_2HAsO_4 all have neutral reaction. NaSH and organic bases are neutral	Luck, *Zeits. anal. Chem.*, **16**, 322. Long, *Chem. News*, **51**, 160, and *Draper*, **55**, 133; *Zeits. anal. Chem.*, 1888, 155. Werder, *Amer. Chem. Journ.*, **3**, No. 1. Léger, *Journ. Pharm. Chim.*, 1885, **6**, 5th series. Thomson, *J. S. C. I.*, **6**, 175
Methyl orange	Water	1 : 1,000	0·1 to 0·2	Cold to blood heat	Sulphuric, hydrochloric, nitric, sulphurous, phosphoric, arsenic and chromic acids, soda, borax, sodium aluminate, organic bases, sulphites, sodium silicate, alkaline sulphides, alkaline fatty acid salts, alkaline acetates, etc.		Nitrous, carbonic, arsenious, silicic, boric, oxalic, acetic and organic acids, alkali in the presence of alumina	Alkalis	1 c.c. $\frac{N}{100}$ HCl	Excess of indicator to be avoided. $KHCrO_4$, $NaHSO_3$, NaH_2PO_4, NaH_2AsO_4, H_3BO_3 are neutral; CO_2 without action; Na_2HPO_4, Na_2HAsO_4 are alkaline. Hyposulphurous acid without action; nitrous acid decomposes the indicator	Lunge, *Chem. Ind.*, 1881, 348. Dammer, *Lexicon der Verfälschungen*, 1887, 821. *Dingl. Polyt. Journ.*, **250**, 530. *Zeits. angew. Chem.*, 1890, 299. *Dingl. Polyt. Journ.*, **251**, 40. On the Theory of Methyl Orange as Indicator, see Küster, 68. *Versammlung der Ges. deutscher Naturforscher und Aerzte in Frankfurt-am-M.*, Sept. 21-26, 1896
Ethyl orange	30 per cent. alcohol	1 : 400	0·2	Cold to blood heat	Similar to methyl orange; used by few chemists		As methyl orange	Alkalis	3 c.c. $\frac{N}{10}$ HCl	Not as sensitive as methyl orange	
Tropaeolin OO	30 per cent. alcohol or water	1 : 400 1 : 2,000	0·2 4	Cold	Mineral acids	Oxalic acid	Organic acids	Acids	2·5 c.c. $\frac{N}{10}$ HCl	Little used; similar to methyl orange, but much less sensitive; CO_2 and bicarbonates without action	Von Miller, *Ber.*, **11**, 460; *Zeits. anal. Chem.*, **17**, 474
Phenacetolin	Alcohol	1 : 200	0·2	Cold or warm	Alkaline carbonates, alkaline earths, calcium carbonate	Alkaline sulphides, ammonia	Organic acids, calcium carbonate in milk of lime	Acids	0·1 c.c. $\frac{N}{10}$ KOH	Unreliable indicator. Nitrites are neutral	Degener, *Zeits. Zuckerind*, 1881, 357; *Chem. Zeit.*, 1887, 591

[*To face page* 4.

TABLE I.—*continued.*

Indicator.	Solvent.	Strength of Solution.	Used for 100 c.c. of Liquid.	Temperature.	Suitable for the Titration of	Less Suitable for	Unsuitable for the Titration of	The Influence Predominates of	Required to give a Distinct Reaction.	Reactions and Remarks.	Literature.
			c.c.								
Poirrier's Blue	Water	1 200	0·1	Hot	Caustic alkali in presence of carbonate, CO_2 in presence of bicarbonate, bicarbonate in presence of normal carbonate		In the presence of large quantities of carbonates	Acids	0·5 c.c. $\frac{N}{100}$ HCl	K_2CrO_4, Na_2HPO_4. Na_2HAsO_4, borax, phenol, all react acid; is delicate reagent for acids. Addition of 20 to 30 c.c. of alcohol sharpens the reaction	Engel and Ville, *Comptes rend.*, **100**, 1073; *Bull. Soc. Chim.*, 1885, 17. Lunge, *Ber.*, **18**, 3,290
Congo Red	30 per cent. alcohol	1 : 100	0·1	Cold or warm	Detection of free H_2SO_4 in alum and in organic acids	Aniline, toluidine	Caustic soda, in the presence of CO_2, sulphates, chlorides, nitrates and ammonium salts	Mineral acids	0·7 c.c. $\frac{N}{100}$ HCl	An indicator of doubtful value. Alum is without action, 0·002 per cent. of free acid gives a reaction	Williamson and Smith, *J. S. C. I.*, **5**, 73. Witt, *Ber.*, **19**, 1,719
Rosolic acid	60 per cent. alcohol	1 : 100	0·5	Cold or warm	Mineral acids in artificial light	Caustic alkalies	Ammonia, organic acids	Acids	0·7 c.c. $\frac{N}{100}$ HCl	Unaffected by artificial light. Nitrites are neutral; similar to corallin but better	Kolbe and Schmitt, *Annalen*, **119**, 169
Corallin	Water	1 : 100	0·5		Similar to rosolic acid		Ditto	Acids	0·6 c.c. $\frac{N}{100}$ HCl	Ditto	Ditto
Carminic acid	Water	1 : 100	0·5		Similar to cochineal tincture			Alkalis	0·7 c.c. $\frac{N}{100}$ HCl	Superfluous; less useful than cochineal	
Cochineal	Water	1 : 80	0·5	Warm	Carbonates of the earths, chalk, sulphides		Alkalis in the presence of alumina, organic acids, in the presence of iron and alumina	Alkalis	3 c.c. $\frac{N}{100}$ HCl	Similar to methyl orange; H_2S and CO_2, have not so much influence as on litmus. $NaHSO_3$ and NaH_2PO_4 are neutral, ammonium salts no action. Good results in artificial light	Luckow, *Journ. prakt. Chem.*, **84**, 424; *Zeits. anal. Chem.*, **1**, 386
Lacmoid	20 per cent. alcohol	1 : 200	0·1		Alkalis, aluminates, calcium and magnesium bicarbonates, borax, alkaline silicates, alkaline aluminates (+ Al_2O_3)	Alkaline sulphites (lacmoid paper), sodium sulphide	Organic acids	Alkalis		Similar to methyl orange. Damaged by light. Bad by artificial light. $NaHCrO_4$, NaH_2PO_4, NaH_2AsO_4, $NaHSO_3$ are neutral. Na_2CrO_4, Na_2HPO_4, Na_2HAsO_4, and Na_2SO are alkaline. Sulphates and chlorides of the heavy metals neutral, H_2S destroys the indicator	Draper, *Chem. News*, **51**, 206. Thomson, *Chem. News*, **52**, 18. Traub and Hock, *Ber.*, **24**, 2,615. Traub, *Arch. Pharm.*, **23**, 27. Forster, *Zeits. angew. Chem.*, 1890, 163
Turmeric	Water				Organic acids, ammonia		Sensitive to CO_2	Alkalis		Salts increase sensitiveness. K_2CrO_4 strongly alkaline. Very sensitive indicator	Storch, *Ber. Chem. Ind. Oest.*, **9**, 95. Storch, *Zeits. anal. Chem.*, 1888, 43 and 60
Indigotin sulphonic acid					Free alkali in the presence of carbonate					Inaccurate, according to Lunge	Engel and Ville, *Comptes rend.*, **100**, 1,073. Lunge, *Arch. Pharm.*, **23**, 243

TABLE II.—*Solutions and Reagents in General Use.*

Name.	Preparation.	Use.	Remarks and Literature.
Fehling's solution	(*a*) 69·278 grms. crystallised copper sulphate dissolved to 1,000 c.c.; (*b*) 173 grms. Rochelle salt and 51·6 grms. caustic soda dissolved to 1,000 c.c.	Equal volumes of (*a*) and (*b*) are mixed before use and boiled with the solution to be tested for dextrose, the presence of which is indicated by a precipitate of cuprous oxide. Qualitative and quantitative	Stammer, *Lehrbuch der Zuckerfabrik.* Von Lippmann, *Die Zuckerarten.* Fresenius, *Quant. Anal.*, II., 462
Nessler's reagent	13 grms. of mercuric chloride are dissolved in 800 c.c. of hot water, 35 grms. of potassium iodide are gradually added until the precipitate redissolves, mercuric chloride solution is then again added drop by drop until a precipitate just remains; 160 grms. of caustic potash are then dissolved in the liquid, which is made up to 1,000 c.c., and the clear yellow solution poured off from the deposit	Used in the detection and colorimetric estimation of ammonia in neutral or alkaline solution	In regard to the colorimetric estimation of NH_3, see under *Water*, Ammonia. J. König, *Chem. Zeit.*, 1897, 599; *J. S. C. I.*, 1897, 936
Starch solution	(*a*) 5 grms. of starch are mixed with a little water and gradually introduced into 1,000 c.c. of boiling water, the liquid is boiled for several minutes, allowed to settle and filtered; (*b*) or soluble starch is used, made (Zulkowsky) by heating starch with glycerin and precipitating by absolute alcohol	Several c.c. are added to the solution in iodometric estimations. Free iodine colours the starch blue	To be kept in a cool place. Is useless when mould is perceptible. To preserve the solution salt may be added, or the whole may be pasteurised and covered with paraffin. Soluble starch must always be kept moist
Starch and potassium iodide solution	Add a few crystals of potassium iodide to the above starch solution	A reagent for nitrous acid, free chlorine, etc. Frequently used as potassium iodide starch paper	To be used with discretion, since ferric chloride also gives the reaction
Starch and zinc iodide solution	4 grms. of starch are ground and gradually added to a boiling solution of 20 grms. of zinc chloride in 100 c.c. of water; after heating some time, until almost clear, the solution is diluted, 2 grms. of zinc iodide added and the volume made up to 1,000 c.c.	As starch and potassium iodide solution	Should give no blue coloration with dilute sulphuric acid. Must be kept in the dark
Uranium acetate solution	About 35 grms. of uranium acetate are dissolved in water containing acetic acid, the solution made up to 1,000 c.c., allowed to stand several days, filtered and standardised against a solution of phosphoric acid	Used in the estimation of phosphoric acid	Other salts of uranium may be used, *e.g.*, the nitrate
Iron ammonium alum	6 parts of ferrous sulphate are dissolved in water. 2 parts of sulphuric acid are added, the solution heated with nitric acid until ferrous salt can no longer be detected by potassium ferricyanide, then 6 parts of ammonium sulphate are added and the solution evaporated to crystallisation, when the compound $Fe_2(NH_4)_2(SO_4)_4 . 24H_2O$ separates	0·5 c.c. of a cold saturated aqueous solution are added in titrating chlorine and copper by Volhard's method	Also a commercial article. J. König recommends iron potassium alum (a solution of 0·898 grm. in 1,000 c.c. contains 0·0001 grm. iron in 1 c.c.)
Potassium sulphocyanide solution	1 grm. dissolved in 10 c.c. of water	An extremely delicate reagent for ferric salts; used in colorimetric estimations of iron	J. Konig, *Chem. Zeit.*, 1897, 599; *J. S. C. I.*, 1897, 936
Schweitzer's reagent	Solution of basic copper sulphate or copper hydroxide in ammonia	A solvent for cellulose, cotton and silk	See under *Textile Fibres*

TABLE II.—*continued.*

Name.	Preparation.	Use.	Remarks and Literature.
Millon's reagent	10 grms. mercury + 25 grms. nitric acid (1·185) + 25 c.c. water, dissolved lukewarm; this solution mixed with a solution of 10 grms. mercury in 22 grms. nitric acid (1·25 – 1·3 sp. gr.) made on the water-bath	Reagent for albumins	Nickel, *Zeits. anal. Chem.*, 1889, 245 (Hoffmann's reagent, Plugge's reagent)
Dobbin's reagent	Mercuric chloride solution is added to a solution of 5 grms. of potassium iodide until a permanent precipitate just appears; the solution is filtered, 1 grm. of ammonium chloride is added and then dilute caustic soda until a permanent precipitate again forms, the liquid is filtered and made up to 1,000 c.c.	Reagent for traces of fixed caustic alkalies (in soda, soap, etc.)	*Chem. Zeit. Rep.*, 1890, 136
Diphenylamine solution	2 grms. of diphenylamine are dissolved in 100 c.c. of hot dilute sulphuric acid (1 : 3), 300 c.c. of strong sulphuric acid (1·84) are then added	Extremely sensitive reagent for traces of nitrates, nitric acid, peroxides, free chlorine	A few drops of the solution to be tested are added to about 20 drops of the reagent, the mixture is shaken for 1 minute. Oxalic acid must be absent
m-phenylenediamine solution	5 grms. are dissolved in water, sulphuric acid is added to acid reaction and the solution made up to 1,000 c.c.	Reagent for nitrous acid	Useful when iron salts prevent the employment of starch and potassium iodide solution
Stannous chloride solution	1 part of crystallised stannous chloride dissolved in 2 parts of hydrochloric acid (1·19)	Bettendorf's reagent for arsenic	*Pharm. Zeit.*, 1891, 167
Alkaline lead solution	1 part of lead acetate is dissolved in 10 parts of distilled water and caustic soda added until the precipitate just redissolves	Reagent for sulphuretted hydrogen	See under *Textile Fibres*
Potassium bichromate solution	A solution containing 3·874 grms. of pure fused potassium bichromate in 1,000 c.c.	To control the strength of standard sodium thiosulphate solution	20 c.c. of this solution liberate 0·2 grm. of iodine (in sulphuric acid solution, containing 1 to 2 grms. of potassium iodide), which is titrated with sodium thiosulphate solution
Sodium nitroprusside solution	1 part of sodium nitroprusside dissolved in 50 parts of water	Delicate reagent for sulphuretted hydrogen and alkaline sulphides	
Indigo solution	Indigotin dissolved in fuming sulphuric acid, and the solution diluted so that 10 c.c. = 0·001 grm. N_2O_5	Estimation of nitric acid and nitrates, estimation of oxygen in water	Chlorates and free chlorine disturb the reaction. See page 1
Potassium nitrate solution	A solution containing 0·1872 grm. of pure potassium nitrate in 1,000 c.c.	In standardising the above indigo solution, 10 c.c. of potassium nitrate solution contain 0·001 grm. N_2O_5	See estimation of nitric acid, under *Water*
Barium chloride solution	A solution containing 0·523 grm. of crystallised Ba Cl_2. $2H_2O$ in 1,000 c.c.	See determination of hardness of water	100 c.c. = 12 degrees (German) of hardness
Ammonium molybdate solution	1 part of molybdic acid dissolved in 4 parts of ammonia (8 per cent. NH_3) and the solution poured into 15 parts of nitric acid (sp. gr. 1·2); or 150 grms. of pure ammonium molybdate dissolved, by heating, in 1,000 c.c. of water, and mixed with 1,000 c.c. of nitric acid (1·2). Allow to stand for several days in a moderately warm place, and, if necessary, pour off from the deposit	Used in the detection and estimation of phosphoric acid	To be kept in the dark. Fresenius, *Quant. Anal.*, II., 540

[*To face page* 4.

CHAPTER II.

PRIMARY MATERIALS.

WATER.

F. Tiemann und A. Gärtner, *Die chemische und mikroskopisch-bakteriologische Untersuchung des Wassers.*
F. Fischer, *Chemische Technologie des Wassers.*

It is sufficiently well known that water plays an all-important part in dyeing. Now the iron, then the lime, or again the sulphuric acid has a disturbing action. The essential characteristics of water required for household and technical purposes are quite different. For the former purpose even traces of ammonia and nitrous acid make a water appear at least suspicious, and, on the other hand, a certain quantity of iron and a considerable degree of hardness are quite harmless and even to some extent impart a pleasant flavour. For technical purposes traces of ammonia, etc., are quite unimportant; the principal attention must be given to the hardness and iron content. The hardness of water is known to produce boiler scale, which causes loss of heat, renders possible boiler explosions and in any case shortens the life of the boiler. Iron on its part injures the dyes and causes spots; it is especially harmful in Turkey red dyeing and in bleaching. Definite limits for the permissible amount of impurities in water for technical or household use cannot be laid down; the whole of the circumstances must be taken into consideration in deciding whether a water may be used, how and to what extent it is to be purified, etc. Purification is naturally avoided as far as possible, since it is a costly process: a good system applied to an average water of 10° of hardness (German) costs about 1 farthing per cubic metre.

Appearance.—Clear or turbid, colourless or coloured. Compare in a glass tube 70 cm. long, 20 mm. wide, with distilled water.

Smell.—Examine at 40° to 50° C. (coal gas, sulphuretted hydrogen).

Taste.—Examine at 15° to 20° C.

Reaction.—Litmus and turmeric paper, cold and hot.

Ammonia.—Qualitatively, by means of Nessler's reagent (which see). Quantitatively, by the colorimetric process, in comparison with an ammonium chloride solution containing 3·147 grammes per litre, 1 c.c. of which contains 1 milligramme of ammonia (NH_3) (J. König, *Chem. Zeit.*, 1897, 599; *J. S. C. I.*, 1897, 936).[1]

Nitrous Acid.—Qualitatively, by means of potassium iodide and starch solution; in the presence of iron salts, by means of *m*-phenylenediamine solution. Quantitatively by a colorimetric process against a solution containing a definite quantity of N_2O_3 (0·406 gramme silver nitrite with excess of sodium chloride in 1,000 c.c.; the filtered solution contains 0·1 milligramme N_2O_3 in 1 c.c.) (J. König, *loc. cit.*).

Carbonic Acid.—Detected by lime water.

Sulphuretted Hydrogen.—Detected by means of lead acetate paper.

Lead and other heavy metals.—Detected by means of sulphuretted hydrogen water.

Nitric Acid.—Qualitatively, by means of diphenylamine solution. Quantitatively, see below.

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[1] The colorimeter recently designed for this purpose for the estimation of NH_3, N_2O_3 and Fe_2O_3, is sold by Dr. R. Muencke, Berlin, N.W.

Hardness of Water.—This property is variously expressed in different countries.

English scale, 1° = 1 grain $CaCO_3$ in 1 gallon of water.
German ,, 1° = 1 part CaO in 100,000 parts of water.
French ,, 1° = 1 part $CaCO_3$ in 100,000 parts of water.

English.	German.	French.
1°	0·56°	0·70°
1·79°	1°	1·25°
1·43°	0·8°	1°

A distinction is made between "total" and "permanent" hardness; when the expression "hardness" is not qualified, total hardness is always understood. Total hardness is the hardness of the untreated water; permanent hardness is that of the water after boiling ten to fifteen minutes, replacing the evaporated water, and filtering. The difference is known as temporary hardness. Both total and permanent hardness are determined in the same manner:—

1. Gravimetric estimations of lime and magnesia, calculation of the magnesia into the equivalent quantity of lime; the sum, expressed as parts of CaO in 100,000 parts of water, gives the hardness on the German scale (see p. 10).

2. Acidimetric estimation: 100 to 200 c.c. of water are evaporated with excess of sodium carbonate to 25 c.c., the resulting alkaline earth carbonates are filtered off, washed to neutral reaction, and titrated together with the filter paper in a porcelain dish with $\frac{1}{10}$ N nitric acid; 1 c.c. $\frac{1}{10}$ N acid = 0·005 gramme $CaCO_3$ = 0·0028 gramme CaO.

3. Titration with a standard soap solution (the methods of Faisst, Knauss and Clark).

(*a*) 0·523 gramme of pure crystallised barium chloride is dissolved and the solution made up to 1,000 c.c.; the quantity of barium chloride contained in 100 c.c. of this solution corresponds to 12° of hardness (German).

(*b*) 20 grammes of pure soap are dissolved in 1,000 c.c. of alcohol (56 per cent. by volume).

Solution (*b*) is now standardised against (*a*): 100 c.c. of the latter are brought into a stoppered bottle of about 200 c.c. capacity, the soap solution is gradually added from a burette until, after violent shaking, the foam remains for five minutes. The soap solution is then diluted with 56 per cent. alcohol, in accordance with the volume used in the experiment, so that 45 c.c. exactly correspond to 100 c.c. of the barium chloride solution. Now 100 c.c. of the water under examination are titrated with this soap solution in the same manner as the 100 c.c. of barium chloride solution, *i.e.*, the soap solution is gradually run from a burette into 100 c.c. of the water, with violent shaking, until the froth endures for five minutes. The hardness of the water, which is not proportional to the soap used, is obtained from the table on p. 9. In case more than 45 c.c. of the soap solution are required, the water under examination must be proportionately diluted with distilled water.

Residue.—100 to 250 c.c. of the water are evaporated in a platinum or porcelain dish on the water-bath, the residue dried at 105° C., weighed, and calculated to 100,000 parts. The residue may be ignited (see *Organic Matter*) and be used in the estimation of iron.

Chlorine.—25, 50 or 100 c.c. of the water are titrated by $\frac{1}{10}$ N silver nitrate solution, with neutral potassium chromate as indicator, until a brown coloration just occurs. The result is calculated to 100 litres. 1 c.c. $\frac{1}{10}$ N silver solution = 0·00354 gramme of chlorine.

Nitric Acid (Trommsdorff-Marx).—10 or 25 c.c. of the water are titrated in a small flask with indigo solution until, on the addition of 20 to 100 c.c. of strong sulphuric acid, the liquid just appears slightly green. This estimation must be performed several times, first approximately, then accurately. It is important that the indigo solution be added *first*, and *then* the sulphuric acid (in the first rough experiment the sulphuric acid may be added first and the mixture titrated with indigo solution). When the solution, after shaking with sulphuric acid, still appears yellow, the experiment must be repeated with more indigo solution, until the point is hit at which a green coloration appears on shaking with sulphuric acid (see *Indigo Solution*).

Soap solution c.c.	Hardness degrees (German).	Soap solution c.c.	Hardness degrees (German).	Soap solution c.c.	Hardness degrees (German).
3·4	0·5	18·9	4·5	34·7	8·9
4 2	0·7	19·7	4·7	35·3	9·1
5·0	0·9	20·4	4·9	36·0	9·3
5·8	1·1	21·2	5·1	36·7	9·5
6·6	1·3	21·9	5·3	37·4	9·7
7·4	1·5	22·6	5·5	38·1	9·9
8·2	1·7	23·3	5·7	38·4	10·0
9·0	1·9	24·0	5·9	38·7	10·1
9·8	2·1	24·8	6·1	39·4	10·3
10·5	2·3	25·5	6·3	40·1	10·5
11·3	2·5	26·2	6·5	40·8	10·7
12·1	2·7	26·9	6·7	41·5	10·9
12·8	2·9	27·6	6·9	42·1	11·1
13·6	3·1	28·4	7·1	42·4	11·2
14·3	3·3	29·1	7·3	42·8	11·3
15·1	3·5	29·8	7·5	43·1	11·4
15·9	3·7	30·5	7·7	43·4	11·5
16·6	3·9	31·2	7·9	43·7	11·6
17·4	4·1	31·9	8·1	44·0	11·7
17·8	4·2	32·6	8·3	44·4	11·8
18·1	4·3	33·3	8·5	44·7	11·9
18·5	4·4	34·0	8·7	45·0	12·0

Nitric acid in other salts may also be estimated by this process.

The results are accurate only at great dilutions; the water must be proportionately diluted when more than 3 milligrammes of N_2O_5 are contained in 25 c.c.

Example: For 10 c.c. of water, 10 c.c. of indigo solution are used (7 c.c. of indigo solution = 1 milligramme N_2O_5); 7 : 1 = 10 : x, or x = 1·43. Thus in 10 c.c. of water there is 1·43 milligramme N_2O_5, or in 100 litres of water 14·3 grammes N_2O_5.

Gravimetric Estimations (SiO_2, Fe_2O_3, Al_2O_3, CaO, MgO, SO_3).

Silica.—Two litres of water (I. and II.) are evaporated separately, the residues moistened with a little strong hydrochloric acid, evaporated and heated for two hours at 105° C. in order to render the silica insoluble. The residues, I. and II., are taken up in hot dilute hydrochloric acid, the silica brought on to an ash-free filter paper, which is dried, ignited and the residue calculated as SiO_2 per 100 litres.

Iron and Alumina.—The filtrate from the SiO_2 of litre I. is precipitated with ammonia, heated for some time to drive off the greater part of the ammonia, the precipitate allowed to settle, the liquid decanted off, filtered, the residue washed with hot water, ignited and weighed = $Fe_2O_3 + Al_2O_3$.

Iron.—In the precipitate, or in a residue obtained by evaporating the water, the iron is determined colorimetrically; the alumina is then calculated by difference (iron and aluminium may also be separated, *e.g.*, by treating the hydrochloric acid solution with excess of caustic soda, in which alumina is soluble, but ferric hydroxide insoluble).

Lime.—The filtrate from iron and aluminium is made ammoniacal, ammonium oxalate added, the liquid left for some hours on the water-bath, filtered after twelve hours, the precipitate dried and ignited, finally before the blowpipe to constant weight. It is weighed as CaO and calculated to 100 litres (to check the result it may be converted into $CaSO_4$).

Magnesia.—The filtrate from the lime is allowed to stand for twelve hours in the cold with ammonium chloride, ammonia and sodium phosphate. The precipitate is filtered off, ignited and weighed. The pyrophosphate, $Mg_2P_2O_7$, obtained is now calculated to MgO and reduced to 100 litres.

Sulphuric Acid.—The filtrate from the silica of litre II. is precipitated boiling by excess of barium chloride after addition of hydrochloric acid. The precipitate is filtered off, ignited, weighed, and the barium sulphate obtained calculated to SO_3 per 100 litres.

Carbonic Acid.—The carbonic acid contained in water is not of much importance in dyeing. The carbonates can be determined by direct titration of 500 c.c. of the water with $\frac{1}{10}$ N hydrochloric acid and methyl orange. Allowance must be made for the sensibility correction of the indicator. 1 c.c. of $\frac{1}{10}$ N acid = 0·0022 gramme CO_2.

Free carbonic acid, present along with the carbonates, is best determined by Pettenkofer's method: 100 c.c. of water are mixed in a flask with 3 c.c. of strong barium chloride solution, 2 c.c. of ammonium chloride solution, and 45 c.c. of standardised baryta solution, the flask is well corked, shaken, and allowed to stand until the precipitate has settled. An aliquot portion (50 or 75 c.c.) of the clear solution is titrated with $\frac{1}{10}$ N nitric acid, the result calculated to the whole volume and deducted from the 45 c.c. of baryta solution or the equivalent amount of nitric acid.

Example: 45 c.c. of baryta water are equivalent to 40 c.c. of $\frac{1}{10}$ N nitric acid; 50 c.c. of the clear solution require 10 c.c. of $\frac{1}{10}$ N acid; $40 - 10 \times 3 = 10$ c.c. of $\frac{1}{10}$ N acid per 100 c.c. of water = 0·22 gramme of CO_2 in 1 litre of water.

Organic Matter.—(*a*) Approximately by igniting the residue obtained on evaporation, moistening with ammonium carbonate and again igniting gently. The loss in weight is organic matter.

(*b*) More accurately by titration with potassium permanganate: 100 c.c. of the water (measured in a graduated flask, not by a pipette) are boiled in a 300 c.c. flask with 10 c.c.

of dilute sulphuric acid (1: 4) and 10 or 20 c.c. of $\frac{1}{100}$ N potassium permanganate solution, for exactly ten minutes, reckoning from the commencement of ebullition. The colour must be distinctly red at the conclusion, otherwise more permanganate must be used. The solution is then cooled to 60° to 70° C., decolourised with $\frac{1}{100}$ N oxalic acid (10 or 20 c.c.) and titrated back with $\frac{1}{100}$ N permanganate until the red coloration begins to be permanent.

The result is generally expressed in grammes of permanganate required by 100 litres of water. 1 c.c. of $\frac{1}{100}$ N permanganate = 0·000316 gramme $KMnO_4$ per 100 c.c., or 0·316 gramme per 100 litres of water. The calculation as organic matter is very properly being abandoned, since its value is problematical, in view of the very varying amounts of permanganate required by different organic compounds.

	Organic Carbon.	Organic Nitrogen.	Ammonia.	Nitrogen Trioxide.	Total Nitrogen.	Chlorine.	Hardness.	
							Permanent.	Total.
Rain water	0·07	0·015	0·029	0·003	0·042	0·22	0·2	0·4
River water	0·322	0·032	0·002	0·009	0·042	1·13	0·8	3·0
Spring water	0·061	0·018	0·012	0·495	0·522	5·11	8·8	13·0
Well water	0·056	0·013	0·001	0·386	0·396	2·49	6·2	10·0

If the water contains much nitrite, 1·66 part of permanganate must be deducted for 1 part of N_2O_3. If it contains considerable quantities of ammonia, this must be removed by a preliminary evaporation to about half the volume, the evaporated water being then replaced by distilled.

As a rule, in dyeing, the quantity of organic matter is without importance. Isolated cases have been observed, in which an abnormal quantity of organic matter decomposed certain artificial dyes or brought about reduction in mordanting wool with potassium bichromate (*Leipz. Färberzeit*, 1897, 8, 88).

Average Values.—To how great an extent the composition of water varies with its source will be seen from the figures given in the preceding table, which were obtained by an English commission as the average values of 600 usable waters. The figures represent grammes per 100,000 parts of water.

Water Purification.—Water may be purified by various methods, mechanical and chemical.

The mechanical purification of water is accomplished by allowing it to flow through a number of filtering layers, by which the suspended particles are retained. For example, a Berlin municipal filter is composed as follows: 0·3 m. of clay, 0·15 m. of linen, 0·3 m. of gravel and coarse sand, 0·45 m. of fine sand; the Altona filter contains 2·2 m. of pebbles the size of a fist, 7·5 m. of coarse gravel, 7·5 m. of fine gravel and 0·9 m. of fine sand.

By chemical purification dissolved constituents are wholly or partially eliminated.

1. Lime or lime water, added in proportion to the temporary hardness; excess of lime is to be avoided.

$$Ca(HCO_3)_2 + Ca(OH)_2 = 2H_2O + 2CaCO_3.$$

2. Water glass, especially used for waters with great permanent hardness; for each 1° of hardness and 100 litres of water, 3 grammes of water glass and 3 grammes of calcined soda are used. Since the precipitate settles very slowly the water must be filtered.

3. Soda and soap, in silk-dyeing. The water is boiled and the scum removed.

4. Barium salts. $BaCO_3 + CaSO_4 = BaSO_4 + CaCO_3$. The disadvantage is that barium carbonate is dissolved by free carbonic acid and the water is thus made poisonous.

5. Ammonium oxalate: is poisonous, and, since excess is unavoidable, should not be used.

6. Alum: not suitable, since potassium and ammonium are introduced.

7. Aluminium sulphate: good action on waters which are coloured. Excess increases the permanent hardness.

8. Ferric chloride, ferrous sulphate.

9. Metallic iron: 1 kilo. of iron wire is used to 100,000 litres of water, ferric hydroxide being formed. Not suitable, since filtering is necessary.

In addition to these systematic methods of purifying water, various so-called "boiler fluids" have been proposed and brought on to the market. Generally speaking (1) they make soluble those salts contained in the water which form boiler scale, or (2) cause the scale to be friable and therefore readily removed, thus increasing the life and safety of the boiler. The substances used for this purpose act mechanically or chemically, the former by contact prevent the precipitation or cause the scale to deposit in a less dangerous condition, whilst the latter enter into chemical reactions with the salts in the water. The following list contains the most important anti-incrustators, mixtures of which in the most varied proportions are used:—

Soda, ammonium chloride, barium chloride, stannous chloride, caustic soda, lime, hydrochloric acid, tannic acid, catechu, oak and pine sawdust, slimy substances, potato chips, dextrine, flour, bran, resin, pitch, clay, paraffin, petroleum and numerous other substances.

The water may also be corrected in the dye-bath itself; this is best done with acetic acid, which is added in quantity equivalent to the temporary hardness. For every 2° of temporary hardness 85 to 86 c.c. of commercial acetic acid are added per cubic metre. If too much acetic acid is added, the formation of the lakes of the colouring matters takes place very slowly, and long boiling is necessary. (See also

Francis Wyatt, *Eng. and Mining Journal*, 1895, 60, 220; Nösselt, *Zeits. d. V. d. Ingenieure*, 1895, 39, 991; E. Schleh, *Das Wasser und der Kesselstein*, published by E. Mayer, Aix-la-Chapelle, 1897.)

Technical Examination of the Suitability of a Water.—Whether a water is suitable for use in dyeing and printing for one purpose or another, and to what extent it influences the shades, can be suitably ascertained by making comparative dyeing, etc., trials with distilled water and the water under examination.

For such an experiment 30 to 40 litres of water are generally sufficient, in which cotton, wool or silk is mordanted, dyed, printed, washed, etc. If the shades turn out worse than those obtained with distilled water, then the water must be condemned, otherwise it is to be regarded as suitable. For such experiments, logwood, fustic, vesuvine and safranine, among others, are suitable (see H. Lange, *Färber-Zeit.*, 1891, No. 23, and A. Lehmann, *ibid.*, 1895, No. 34, p. 378).

Effluents.—In the examination of effluents, the corrosive, malodorous and poisonous constituents receive the first attention. Thus, in the first place, the acidity or alkalinity, then the presence or absence of sulphuretted hydrogen, substances producing that gas, and in general substances of bad odour, and finally the presence or absence of poisonous matters (lead, arsenic, aniline etc.), must be ascertained.

As with water for use, the following constituents also serve to characterise an effluent: residue on evaporation, organic matter, suspended matter, lime, magnesia, chlorine, sulphuric acid, ammonia, nitric acid, nitrous acid, phosphoric acid, etc. (*cf.* F. Fischer, *Zeits. angew. Chem.*, 1890, 64; *J. S. C. I.*, 1890, 93, and J. König, *Zeits. angew. Chem.*, 1890, 88).

TEXTILE FIBRES.

Persoz, *Le conditionnement, le titrage et le décreusage de la Soie, suivi de l'examen des autres textiles.* Paris, 1887.

H. Schacht, *Die Prüfung der im Handel vorkommenden Gewebe durch das Mikroskop und durch chemische Reagentien.*

J. Herzfeld, *Die technische Prüfung der Garne und Gewebe.*

Von Höhnel, *Mikroskopie der technisch verwendeten Faserstoffe.*

Detection of:—

Cellulose, wood fibre, lignified fibre.

1. Iodine solution and sulphuric acid mixture.
   (*a*) One gramme of potassium iodide dissolved to 100 c.c., iodine then added to saturation (excess remains at the bottom of the solution).
   (*b*) Three volumes of strong sulphuric acid are slowly added, with cooling, to a mixture of 2 volumes of the purest glycerin and 1 volume of distilled water.

The test is carried out as follows: The fibre under examination is moistened on the slide with a few drops of the above iodine solution, the excess removed after some time by means of filter-paper and one to two drops of solution (*b*) added. Pure cellulose does not swell and is coloured pure blue; lignified fibres are coloured yellow.

Cellulose.

2. Zinc chloride iodine solution. In place of the above two solutions a solution of iodine and zinc chloride may be used, which colours cellulose reddish to bluish violet. A solution of iodine in potassium iodide solution is mixed with strong zinc chloride solution (according to von Höhnel, 1 part of iodine, 5 parts of potassium iodide, 30 parts of zinc chloride and 14 parts of water; or 100 parts of $ZnCl_2$ of sp. gr. 1·82 + 12 parts of H_2O + 6 parts of KI + iodine, until iodine vapours are evolved).

Detection of :—

Lignified fibres.

3. An aqueous solution of indol, followed by the addition of hydrochloric acid (red coloration); aniline sulphate or hydrochloride, followed by dilute hydrochloric acid (golden-yellow coloration); phloroglucinol and hydrochloric acid (red coloration); naphthylamine hydrochloride (orange coloration).

Sections of jute give no colour reactions with these reagents, whilst they are coloured a fine yellow by No. 1 (iodine solution and sulphuric acid).

Cotton and lignified fibres.

4. Ammoniacal copper oxide. Freshly precipitated copper hydroxide is dissolved in strong ammonia, and the solution kept in dark well-stoppered bottles. The solution at once dissolves dry cotton; cellulose, slightly lignified fibres (hemp), swell considerably or dissolve; strongly lignified fibres barely swell.

Animal or vegetable fibre.

5. Naphthol solution, 0·01 gramme of pure fibre is treated with 1 c.c. of water and 2 drops of a 15 to 20 per cent. alcoholic solution of α-naphthol, 1 c.c. of strong sulphuric acid is then added. If a vegetable fibre is present, a deep violet solution is formed on shaking; animal fibres give a yellowish-reddish brown coloration (the fibre remains undissolved). If thymol be used instead of α-naphthol, the solution becomes red.

The following conclusions may be drawn, based upon the coloration and the solubility or insolubility of the fibre.

Violet Coloration.	
The fibre dissolves at once	vegetable fibre alone or mixed with silk.
The fibre partially dissolves	vegetable fibre mixed with wool or perhaps with silk.

Slight or no Coloration.	
The fibre dissolves at once	silk.
The fibre does not dissolve	wool.
The fibre partially dissolves	wool mixed with silk.

Detection of:—

Animal and vegetable fibres.

6. A caustic soda or potash solution of 8 per cent. strength (6 to 7° B. = 1·04 sp. gr.) dissolves animal fibres, whilst vegetable fibres remain almost unattacked. Wool dissolves in five minutes, silk in ten to fifteen minutes at water-bath temperatures.

The following method for estimating cotton in woollen fabrics is official in Germany: to 5 grammes of woollen yarn contained in a beaker of 1 litre capacity are added 200 c.c. of 10 per cent. caustic soda solution; the liquid is slowly (in about twenty minutes) brought to the boil over a small flame and then maintained gently boiling for fifteen minutes longer. The wool is then completely dissolved (dressed woollen yarn must previously be treated with 3 per cent. hydrochloric acid and washed with hot water). When the wool is dissolved, the liquid is filtered through a weighed Gooch crucible, which is dried at a gentle heat, allowed to stand a short time in the air, and weighed. The increase in weight of the crucible gives the weight of the cotton fibre.

Wool and cotton.

7. Rosaniline solution. If wool and cotton be dipped for some seconds in a warm or hot colourless solution of rosaniline containing ammonia, and then washed in the cold, the wool becomes red as soon as all the alkali is removed, whilst the cotton remains colourless. Silk behaves like wool; linen, jute, etc., as cotton. The colourless rosaniline solution is obtained by adding caustic soda or ammonia to a boiling solution of magenta and filtering.
8. Nitric acid. Boiling dilute nitric acid colours wool yellow (silk to a less extent), whilst vegetable fibres (cotton, flax, hemp, etc.) remain colourless.

Detection of :—

Silk, wool, cotton.

9. Nitric and sulphuric acids. Silk (and goats' wool) when left for fifteen minutes in a nitrating mixture (equal volumes of strong nitric and sulphuric acids) is completely dissolved; wool is coloured yellow to yellowish brown; vegetable fibres, on the contrary, are altered neither in colour nor structure (guncotton).

Phenomena of combustion.

10. Animal fibres burn slowly, since they contain nitrogen, and give off a smell peculiar to many nitrogenous substances (horn, hair, hoofs, etc.). They leave a residue of carbon, which burns with difficulty, and relatively much ash. The volatile products of combustion redden moist turmeric paper. Vegetable fibres burn readily, producing an empyreumatic acid odour (somewhat like paper), they give little ash and the vapours redden moist neutral litmus paper.

Behaviour towards various reagents.

11. (*a*) Silk remains undissolved by sulphuric acid for only a few moments, whilst wool remains longer undissolved. Hence a process for the quantitative separation of silk and wool: dilute the solution and filter.

(*b*) Silk and vegetable fibres are not coloured by a solution of litharge in caustic soda, whilst wool, on account of the sulphur it contains, is coloured brown.

(*c*) Ammoniacal copper oxide solution dissolves silk, wool remains undissolved.

(*d*) A solution of wool in caustic potash gives a violet coloration with sodium nitroprusside.

(*e*) Wool dissolves in 10 per cent. caustic soda on the water-bath within five minutes, whilst silk requires ten to fifteen minutes for complete solution.

Detection of :—

Cotton and linen.

12. (*a*) Kindl's test. The fibre, freed from dressing, is placed in strong sulphuric acid for half a minute to two minutes, according to its thickness, it is washed with water, gently rubbed with the fingers, placed in dilute ammonia and dried. Any cotton present is converted by the acid into a jelly, which is removed by the rubbing and washing; linen, on the contrary, is little altered.

(*b*) The fibre under examination is dipped in olive oil and the excess of oil removed by gentle pressure between filter paper. Linen acquires a gelatinous, translucent appearance (somewhat similar to that of oiled paper), whilst cotton remains unaltered. Hence on a dark background the linen fibres appear dark, the cotton fibres light (Frankenheim and Leykauf).

(*c*) Rosolic acid test. Linen becomes rose-coloured when treated with an alcoholic solution of rosolic acid and then with strong soda solution; cotton is discoloured.

Detection of:—

Quantitative estimation of silk, wool and cotton.

13. Zinc chloride solution (Rémont). The purified fibre (about 2 grammes), freed from dye, is

(*a*) placed in a boiling solution of basic zinc chloride (sp. gr. 1·69 to 1·70), left fifteen minutes on the water-bath and washed until the washings no longer contain zinc. The silk is dissolved, its amount is estimated by the loss in weight. The zinc chloride solution is obtained by heating together 1,000 parts of zinc chloride, 850 parts of water and 40 parts of zinc oxide until solution is complete.

(*b*) A further quantity of 2 grammes is immersed in 60 to 80 c.c. of caustic soda solution (1·5 per cent.) which is gently boiled for fifteen minutes, the fibre is carefully washed, dried and weighed; the loss in weight = wool.

(*c*) The remainder is vegetable fibre; 5 per cent. must be added, since the original has been somewhat decomposed, and the same amount must be deducted from the percentage of wool.

Shoddy.

14. Artificial wool or shoddy is a mixture of used wool and wool waste with more or less unused wool, used silk, silk waste, flax and cotton. Shoddy is more rapidly swollen and dissolved by alkalis than new wool. The vegetable fibres are estimated according to 13 (*b*), by removing the woollen fibre. The silk is estimated according to 13 (*a*), or by means of strong sulphuric acid, in which the silk dissolves rapidly, the wool slowly; the loss in weight = silk. The approximate composition of shoddy can also be well estimated microscopically (see Plate I.). Shoddy is distinguished under the microscope by the irregularity and varying diameter (sudden contractions and swellings) of the fibres, the want of scales, the shortness of the individual hairs and difference of colour.

Detection of :—

Genuine and tussur silk.

15. In order to distinguish mulberry silk from the wild or tussur silk, Persoz boils for one minute with a zinc chloride solution of 45° B.; genuine mulberry silk is dissolved, tussur silk is hardly attacked.

Artificial silk.

16. Artificial silk is composed essentially of nitrocellulose, natural silk of albuminous matters.
    - (*a*) Chardonnet nitrates cellulose to the octonitrate, which is then dissolved in ether-alcohol.
    - (*b*) Du Vivier dissolves trinitrocellulose in glacial acetic acid.
    - (*c*) Lehner dissolves nitrocellulose in methyl alcohol-ether or ether-sulphuric acid, and adds natural silk waste, dissolved in glacial acetic acid.
    - (a_1) Chardonnet coagulates with a mixture of alcohol and water or with dilute sulphuric acid.
    - (b_1) Du Vivier uses a secret fluid.
    - (c_1) Lehner coagulates with a mixture of turpentine oil, chloroform and oil of juniper.
    - (a_2) Chardonnet removes the danger of inflammability ("denitrates") by means of dilute nitric acid (sp. gr. 1·32), ferrous chloride, ammonium phosphate, etc.
    - (b_2) Du Vivier denitrates by the addition of 20 per cent. of fish glue and 10 per cent. of gutta-percha, followed by a complicated treatment.
    - (c_2) Lehner dilutes with fire-proofing salts such as sodium acetate, etc.
    - (a_3) Chardonnet's silk is lustrous, pliable and possesses the peculiar "feel" of boiled-off silk.
    - (b_3) Du Vivier's silk is more brittle, but very white and more lustrous than natural silk.
    - (c_3) Lehner's silk has only just recently been brought into commerce.

Detection of :—

Artificial and natural silk.

17. The artificial silks can be distinguished from natural silk by means of the following reactions :—
    (*a*) Artificial silk dissolves in alkalis with a yellow, natural silk with a white colour.
    (*b*) Artificial silk is insoluble in an alkaline copper solution containing glycerin, in which natural silk dissolves at the ordinary temperature. Ten grammes of copper sulphate are dissolved in 100 c.c. of water and 5 grammes of pure glycerin added, caustic alkali is then added until the precipitate redissolves. This solution not only renders possible the detection of artificial silk, but also a complete separation and quantitative estimation of artificial silk in mixtures with natural silk.
    (*c*) Artificial silk gives the nitric acid reaction with diphenylamine and brucine, natural silk does not (*Chem. News*, 1897, 2, 121).

Reagents (for following table).

18. Aniline sulphate, in 1 per cent. aqueous solution, colours lignified cells, according to the extent of lignification, pale yellow to golden yellow.
19. Phloroglucinol, in 0·5 per cent. aqueous solution, indicates lignified cells by a slight reddish to a deep red coloration, when the fibre has been previously moistened with hydrochloric acid.
20. Iodine with sulphuric acid. Two scales of iodine are moistened in a flask with 5 to 6 drops of alcohol, and then so much water added that a slightly wine-yellow solution remains. The object to be examined is

moistened with dilute sulphuric acid (1:2) and then with the iodine solution, or *vice versa.*

21. A 5 per cent. alcoholic solution of magenta.
22. A 5 per cent. aqueous solution of lead acetate.
23. Saturated aqueous solution of picric acid.
24. Basic zinc chloride solution as 13(*a*).

By means of these reagents the following course of examination is conducted (Pinchon):—

10 per cent. solution of caustic soda or potash.	All dissolves.	Zinc chloride (24 or 13(*a*)) dissolves all in the cold.	The solution is *not* blackened on the addition of lead acetate, 22.			Silk.
		Zinc chloride dissolves partially.	The soluble part is not blackened by 22. The insoluble part is turned brown or black by 22.			Silk and Wool.
		Zinc chloride dissolves nothing.	The mass is blackened by lead acetate, 22.			Wool.
	Remains undissolved.	24 dissolves nothing.	Chlorine water or ammonia colours the fibre reddish brown.	Fibre coloured red by nitric acid.		New Zealand Hemp.
			Chlorine water or ammonia does not colour the fibre.	21 colours the fibre permanently.	20 colours it yellow.	Hemp.
				Caustic potash colours the fibre yellow.	20 colours it blue.	Flax.
				The coloration by 21 is not permanent, but can be washed out. Caustic potash does not colour the fibre yellow.		Cotton.
	Dissolves partially.	24 dissolves partially.	22 blackens a portion.	Caustic potash partially dissolves the portion insoluble in 24; the residual fibres dissolve in ammoniacal copper oxide, 4.		Mixture of Wool, Silk and Cotton.
			22 colours nothing.	23 colours a portion yellow, the rest remains white.		Silk and Cotton.
		24 dissolves nothing.	Nitric acid colours a portion yellow, the rest remains white.			Wool and Cotton.

In addition to the chemical examination of the textile fibres, which now are numbered by hundreds, microscopic examination is not to be neglected. The advantage of the latter is that the fibre, whether dyed or not, generally does not require the treatment which is a necessary preliminary to the chemical examination—removal of dressing and dye. It is sufficient to moisten the single, well-isolated fibre with glycerin or water, or to prepare sections of it, and at once to examine under the microscope. On the other hand, a certain amount of practice is requisite; a course of examination of the known fibres must be followed, and their characteristics learnt. However, without any great amount of practice, it is possible to recognise at least the more important fibres, by making use of a proper collection of samples of fibres for comparison.

The author again wishes to draw attention to the splendid work of von Höhnel, already mentioned, *Mikroskopie der technischen verwendeten Faserstoffe;* he also directs attention to Plate I. at the end of the book, a reproduction of the microscopic appearances of the most important fibres, taken from von Höhnel's book.

CHAPTER III.

INORGANIC MATERIALS.

HYDROCHLORIC ACID.

THE strength of hydrochloric acid may be estimated by means of a determination of the specific gravity, or better—since a small proportion of sulphuric acid increases the density considerably—by volumetric processes. Fifty grammes are diluted to 1,000 c.c. and 100 c.c. titrated with normal caustic soda; 1 c.c. of normal soda = 0·0364 gramme of hydrochloric acid.

When other acids (sulphuric) are not to be determined as hydrochloric acid, the proportion of pure hydrochloric acid, or hydrochloric acid + chlorides, may be determined by Volhard's process: 20 to 25 grammes of the acid are diluted to 1,000 c.c., excess of $\frac{1}{10}$ N silver solution is added to 20 or 25 c.c of the dilute acid, after adding nitric acid and a few c.c. of a strong solution of iron alum. The excess of silver is titrated by $\frac{1}{10}$ N ammonium sulphocyanide solution. As soon as all the silver is precipitated as sulphocyanide, the reaction between the sulphocyanide and iron alum occurs, and is recognised by the brownish coloration. The volume of silver solution added, *minus* the sulphocyanide solution used, gives the number of c.c. of silver nitrate solution required to combine with the chlorine. 1 c.c. of $\frac{1}{10}$ N silver solution = 0·00354 gramme of Cl = 0·00364 gramme of HCl. If chlorides be present in addition to the free acid, they must be estimated, in the residue left on evaporation, by means of silver nitrate and potassium chromate; the amount of hydrochloric acid found must be corrected accordingly. The difference between total acid (as found by titration with caustic soda) and hydrochloric acid represents the foreign acids. A simpler process, when the total acids are required, is as follows: the hydrochloric acid solution, after titration with caustic soda, which is now a neutral chloride solution, is titrated directly with silver nitrate and potassium chromate. 1 c.c. of $\frac{1}{10}$ N silver solution = 0·00364 gramme of HCl. It should not be necessary to state that the whole volume of hydrochloric acid neutralised by soda is not titrated by silver nitrate, but, since decinormal silver solution is used, an aliquot part of the liquid only is titrated, or a smaller quantity of hydrochloric acid is taken specially for this purpose.

The presence of free chlorine and nitric acid is detected by means of diphenylamine (which see), free chlorine also by the bleaching action on litmus paper. Other impurities are principally sulphuric acid, iron, arsenic, other heavy metals, alkali salts, etc.; a portion of the acid is evaporated, the residue should not be more than a trace. The acid should not contain much sulphuric acid, in the case of crude hydrochloric acid it should not exceed 1 per cent. (may be estimated by precipitation with barium chloride). For many purposes only a very small quantity of iron is permissible; acid for bleaching may not contain more than 0·03 per cent. (test with potassium sulphocyanide). The iron may conveniently be estimated colorimetrically (see under *Water*) or also gravimetrically and volumetrically (see *Iron Salts*).

Applications.—Generally speaking less used than sulphuric acid, since it always bleaches to some extent, if it contains or produces free chlorine. In bleaching, to "sour" the goods (for this purpose it should not contain more than 0·03 per cent. of iron); to decompose the bleaching powder after bleaching (for this purpose it must be absolutely free from iron); in bleaching with barium peroxide; in aniline black dyeing as aniline hydrochloride (cotton, mixtures of cotton

Specific Gravity and Strength of Hydrochloric Acid (Kolb).

°B	Specific gravity	At 0° C. 100 parts contain HCl.	At 15° C., 100 parts contain parts of			
			HCl.	Acid of 20° B.	Acid of 21° B.	Acid of 22° B.
0	1·000	0·0	0·1	0·3	0·3	0·3
1	1·007	1·4	1·5	4·7	4·4	4·2
2	1·014	2·7	2·9	9·0	8·6	8·1
3	1·022	4·2	4·5	14·1	13·3	12·6
4	1·029	5·5	5·8	18·1	17·1	16·2
5	1·036	6·9	7·3	22·8	21·5	20·4
6	1·044	8·4	8·9	27·8	26·2	24·4
7	1·052	9·9	10·4	32·6	30·7	29·1
8	1·060	11·4	12·0	37·6	35·4	33·6
9	1·067	12·7	13·4	41·9	39·5	37·5
10	1·075	14·2	15·0	46·9	44·2	42·0
11	1·083	15·7	16·5	51·6	48·7	46·2
12	1·091	17·2	18·1	56·7	53·4	50·7
13	1·100	18·9	19·9	62·3	58·7	55·7
14	1·108	20·4	21·5	67·3	63·4	60·2
15	1·116	21·9	23·1	72·3	68·1	64·7
16	1·125	23·6	24·8	77·6	73·2	69·4
17	1·134	25·2	26·6	83·3	78·5	74·5
18	1·143	27·0	28·4	88·9	83·0	79·5
19	1·152	28·7	30·2	94·5	89·0	84·6
19·5	1·157	29·7	31·2	97·7	92·0	87·4
20	1·161	30·4	32·0	100·0	94·4	89·6
20·5	1·166	31·4	33·0	103·3	97·3	92·4
21	1·171	32·3	33·9	106·1	100·0	94·9
21·5	1·175	33·0	34·7	108·6	102·4	97·2
22	1·180	34·1	35·7	111·7	105·3	100·0
22·5	1·185	35·1	36·8	115·2	108·6	103·0
23	1·190	36·1	37·9	118·6	111·8	106·1
23·5	1·195	37·1	39·0	122·0	115·0	109·2
24	1·199	38·0	39·8	124·6	117·4	111·4
24·5	1·205	39·1	41·2	130·0	121·5	115·4
25	1·210	40·2	42·4	132·7	125·0	119·0
25·5	1·212	41·7	42·9	134·3	126·6	120·1

with wool and silk, and recently, experimentally, wool); in blue printing; to remove lime after passing through substances containing lime; in carbonising woollen rags (shoddy); in the manufacture of bleaching powder; in black silk dyeing (also wool-dyeing); in grounding with Prussian blue, etc.

CHLORIDES.

The chlorides of the alkalis and alkaline earths are required to dissolve to a clear solution, have a neutral reaction, be dry and free from foreign metals. Thus, according to circumstances, the solubility, proportion of water and the presence of foreign acids and metals will be investigated. In a detailed examination the halogen and metal may both be estimated. As an example, see the analysis of stannic chloride. In neutral salts the hydrochloric acid may be estimated by means of silver solution, using neutral potassium chromate, K_2CrO_4, as indicator (see *Water* and *Hydrochloric Acid*). Several of the more important chlorides are specially mentioned in the following pages.

Sodium Chloride, Common Salt, $NaCl = 58{\cdot}5$; solubility in cold water = 35 : 100; in hot water = 39·5 : 100.

Applications.—Little used in the textile industries; is added to the bath in dyeing with azo dyestuffs; as a dressing. For the latter purpose it is to be used with caution, since almost all dyes are injured by it, and, being hygroscopic, it gives rises to the formation of "mould spots".

Magnesium Chloride, $MgCl_2 . 6H_2O = 203$; solubility, cold water = 150 : 100; hot water = 367 : 100.

Applications.—It is principally used as a dressing, since it gives the material a damp full feel. However, it should not be used in too large quantities (not more than about 100 grammes per litre), since in the succeeding operations, in singeing in the finishing cylinder, etc., decompositions may occur ($MgCl_2 + H_2O = MgO + 2HCl$) and the material be made rotten. In spite of this, 600 to 700 grammes per kilo. are frequently employed.

Ammonium Chloride, Sal ammoniac, $NH_4Cl = 53{\cdot}4$; solubility, cold water = 37 : 100; hot water = 100 : 100.

This salt should leave only a slight residue on ignition, it should not contain too much water, iron or lime. It is not much used, principally as a hygroscopic substance in dressings, also as an addition to printing colours, and in aniline black dyeing as a carrier of moisture in order to accelerate the oxidation.

Barium Chloride, $BaCl_2 . 2H_2O = 244$; solubility, cold water = 38·4 : 100; hot water = 78·1 : 100.

By passing through a solution of this salt and then through sulphuric acid, large quantities of barium sulphate can be precipitated in the fabric (flannel); this application is restricted owing to the poisonous nature of the compound. It has also been proposed as a water purifier (see *Water*).

Zinc Chloride, $ZnCl_2 = 136$; solubility, cold water = 300 : 100; hot water, melts.

Applications.—Principally used in bleaching paper, also as a mordant for Water Blue and as a double salt of certain commercial dyes (green, methylene blue, etc.). In the manufacture of lakes it is used together with tin, alumina and iron (lakes of the ponceaux, eosines, etc.), also it is added in small quantities (5-10 grammes : 1,000) to dressings, as a preservative, to prevent the formation of mould.

Copper Chloride, $CuCl_2 . 2H_2O = 170{\cdot}5$; solubility, cold water = 60 : 100; hot water, melts.

Copper and ferrous sulphates are the principal impurities (examination similar to that of copper sulphate, which see). It may be used as a good oxidising agent in place of copper sulphate and sulphide (aniline, catechu).

Manganous Chloride, $MnCl_2 . 4H_2O = 198$; solubility, cold water = 150 : 100; hot water = 650 : 100.

This compound has a restricted application in printing certain fashionable colours and as a fixing agent for catechu; also used in the preparation of manganese bistre.

Aluminium Chloride, $Al_2Cl_6.12H_2O$; solubility, cold water = 4 : 1; hot water, melts.

Also occurs commercially as a solution of 20° to 30° B. ("Chloralum"). Its principal application is in carbonising wool (in the better class of goods used at a strength of 3° to 6° B.); also for certain special purposes, *e.g.*, when cotton and wool are woven together and the cotton then removed by carbonisation, so that the skeleton of wool remains.

Stannous Chloride, Tin Crystals, Tin Salt, $SnCl_2.2H_2O$; solubility, cold water = 271 : 100 (with decomposition); hot water decomposes it.

This important salt often comes into commerce chemically pure; it is, however, often adulterated with magnesium sulphate—since the two salts are similar in appearance—also with zinc chloride and sulphate.

Stannous chloride should be analysed quantitatively. There is no really good method; the tin and chlorine may be estimated gravimetrically and the actual stannous chloride volumetrically. All the volumetric processes are based upon the reducing properties of stannous chloride.

Total Tin.—20 grammes of tin crystals are dissolved in 500 c.c. of water acidified with sulphuric acid; in 25 c.c. of the solution the tin is precipitated by sulphuretted hydrogen, the precipitate dried, roasted to stannic oxide with the proper precautions (Fresenius, *Quant. Anal.*, i., 280), weighed, and calculated to metallic tin or stannous chloride; 150 parts of SnO_2 = 118 parts of Sn = 225 parts of $SnCl_2.2H_2O$.

Stannous Chloride.—(*a*) A second 25 c.c. of the solution are titrated by $\frac{1}{5}$ N permanganate after the addition of ferric chloride free from ferrous salt. Stannic chloride and an equivalent quantity of ferrous chloride are formed, the latter being then oxidised by the permanganate to ferric chloride. Boiled water, free from oxygen, must be used (see Fresenius, *Quant. Anal.*, i., 280). 1 c.c. of $\frac{1}{5}$ N potassium permanganate = 0·0225 gramme of $SnCl_2 . 2H_2O$.

(*b*) To 10 c.c. of the solution 50 c.c. of a 10 per cent. solution of Rochelle salt and 50 c.c. of a 10 per cent. solution of sodium bicarbonate are added, and the mixture titrated with $\frac{1}{10}$ N iodine solution, using starch solution as indicator. 1 c.c. of $\frac{1}{10}$ N iodine = 0·01125 gramme of $SnCl_2 . 2H_2O$.

Total and Free Acid.—(*a*) According to Knecht, Rawson and Löwenthal, the total acid may be determined by immediate titration of the solution (20 c.c.) with normal alkali and phenolphthaleïn (methyl orange is better). The separated stannous hydroxide is said not to affect the titration.

(*b*) The acid may be determined more accurately as under *Stannic Chloride* (*a*) and (*b*).

From the amount of tin found the combined acid is calculated, an excess corresponds to free acid, a deficit to basic chloride.

Qualitative Examination.—Pure stannous chloride is soluble in 5 parts of absolute alcohol, almost all impurities and basic salt remaining undissolved. By this means salts of magnesium, zinc, copper and lead, common salt, Glauber's salt, etc., may be found; also an idea is obtained of the proportion of basic chloride, which forms very readily on long keeping: $3SnCl_2 + O + H_2O = 2Sn(OH)Cl + SnCl_4$. The clearer the solution of the salt in alcohol or water, the less basic salt it contains. A commercial product cannot be required to give a completely clear solution.

Applications.—Stannous chloride finds extended use in the following operations: in dyeing wool with flavine and cochineal (to 7½ to 10 per cent. of cochineal, about 3 to 5 per cent. of stannous chloride, with a little tartar, and perhaps also oxalic acid; oxalic acid shades towards yellow, stannous chloride towards blue, cochineal giving a red); in silk-dyeing as a mordant and as fixing agent for catechu; also to make dyes fast to water ($SnCl_2$ was formerly used together with $SnCl_4$); in calico-printing as a discharging agent, since it reduces and destroys the azo dyestuffs; it is also the raw material from which other tin compounds are prepared, *e.g.*, tin acetate.

Stannic Chloride, Tin Perchloride, $SnCl_4 . 5H_2O = 350$. Melts in water; decomposed by much water; contains 33·7 per cent. of tin.

In an exhaustive examination the following estimations are requisite, in order to obtain a complete knowledge of the composition of the substance: total tin, total acid calculated as chlorine, stannous chloride and stannic chloride, foreign acids (sulphuric and nitric), free chlorine, ammonia, alkali salts and foreign metals, metastannic acid.

Total Tin.—25 to 30 grammes of stannic chloride are dissolved to 1 litre; 50 c.c. of the solution are treated with two drops of bromine water (or iodine) in order to oxidise any stannous chloride present, the bromine is expelled by boiling, and the tin precipitated by excess of ammonium nitrate or sodium sulphate. In case the stannic chloride is strongly acid, a few drops of ammonia are added until the solution begins to be turbid, when it is boiled with ammonium nitrate or sodium sulphate (about 20 grammes) for ten to fifteen minutes. The precipitate is allowed to settle, the liquid decanted off, the precipitate filtered, well washed (especially if sodium sulphate was used), dried, ignited and weighed as tin dioxide, SnO_2. Commercial stannic chloride contains 21·5 to 34 per cent. of tin.

Total Acid (T. Goldschmidt).—(*a*) 10 c.c. of liquid stannic chloride are weighed off (or 8 to 9 grammes of the solid salt), and diluted to 100 c.c.; 10 c.c. of the solution are brought into a 100 c.c. flask, diluted with 30 to 40 c.c. of water and then so much $\frac{1}{3}$ N soda solution added, that 0·05 to 0·15 gramme of Na_2CO_3 remains in excess; the liquid is then made up to 100 c.c., well shaken and filtered. (In the case of stannic chloride of about 50° B., 42 c.c. of $\frac{1}{3}$ N soda solution are taken; with solid stannic chloride the necessary quantity of soda solution is calculated from the percentage of tin.) It is absolutely necessary that the soda should be used in such quantity that the excess is at the above degree of dilution, with a larger excess the tin is again partially dissolved and would then be calculated as chlorine; 50 c.c. of the filtrate are now titrated with methyl orange and N or $\frac{1}{5}$ N or $\frac{1}{10}$ N hydrochloric or sulphuric acid; the excess of soda, and thence the soda used, is calculated. If litmus or phenolphthaleïn is used, 10 c.c. of $\frac{1}{2}$ N hydrochloric acid are added to 50 c.c. of the filtrate, the liquid is boiled for five minutes, allowed to cool and titrated. The volume of soda solution used is calculated to chlorine.

Basicity.—The deficiency or excess of chlorine is calculated from the percentages of tin and acid. If there is a deficiency of chlorine, basic salts (tin oxychloride) or stannous chloride (see below) are present; if the chlorine is in excess, free acid is present. The best stannic chloride contains tin and chlorine in exactly equivalent proportions (1Sn to 4Cl).

Example: 10 c.c. of stannic chloride weighed 16·000 grammes. The gravimetric analysis gave 24·25 per cent. of tin, which corresponds to 29·18 per cent. of chlorine. In the experiment 42 c.c. of $\frac{1}{3}$ N soda solution were used; 3·85 c.c. of decinormal acid were required in titrating back. Now $14 - 0{\cdot}385 \times 2 = 13{\cdot}23$ c.c. of N soda were used; $13{\cdot}23 \times 0{\cdot}0355 = 0{\cdot}469665 = 0{\cdot}47$ nearly; $1{\cdot}6 : 0{\cdot}47 = 100 : x$, then $x = 29{\cdot}37$ per cent. of chlorine, or rather total acid calculated as chlorine. Accordingly there is an excess of acid of $29{\cdot}37 - 29{\cdot}18 = 0{\cdot}19$ per cent.

(*b*) The following method is much simpler and more rapid, whilst it is not less accurate than the preceding: 25 c.c. of stannic chloride solution (or 15 to 20 grammes of the solid salt) are dissolved to 250 c.c., 25 c.c. of this solution are mixed in a beaker with hot distilled water, oxidised with one drop of bromine water, and the turbid liquid kept almost at the boiling point on the water-bath for about ten minutes longer. The stannic chloride is quantitatively decomposed into stannic hydroxide and free hydrochloric acid, even though it contains a considerable quantity of free acid. The stannic hydroxide is collected on an ashless filter, washed with hot water until the filtrate is neutral, the precipitate dried, the paper burnt, the whole ignited and weighed as SnO_2. The filtrate is titrated with N caustic soda and phenolphthaleïn, 1 c.c. = 0·0354 gramme of chlorine. The weight of tin dioxide multiplied by 0·7867 gives the weight of metallic tin; the weight of the tin or its percentage, multiplied by 1·2, gives the quantity of chlorine required to form $SnCl_4$. This method is very exact and deserves preference over the preceding.

1 gramme SnO_2 = 0·7867 gramme Sn = 0·7867 × 1·2 gramme Cl, which is required to saturate the tin. The difference between the chlorine found and calculated gives the excess or deficiency of chlorine (see above).

(*c*) The estimation of the free acid by immediate titration of the stannic chloride with normal caustic soda according to Knecht, Rawson and Löwenthal is quite inaccurate and useless; the method of Silbermann (*Färber-Zeit.*, 1897, No. 5) is equally tedious and inaccurate.

The total acid may, however, be approximately estimated by direct titration of the solution of the salt with normal caustic soda and methyl orange as indicator (*not* phenolphthaleïn).

Stannous Chloride.—Qualitatively by means of mercuric chloride. Quantitatively by means of iodine or permanganate (see *Stannous Chloride*).

Nitric Acid, Free Chlorine and *Ammonia* are generally only present in slight traces. They are usually only sought qualitatively, but may be estimated colorimetrically. It must not be forgotten that ferric chloride gives the reaction with potassium iodide starch paper. Nitric acid may be estimated by means of indigo solution, exactly as in water (see *Water* and *Nitric Acid*).

Sulphates of sodium and iron are occasionally fraudulently added in order to increase the specific gravity. If the sulphuric acid reaction indicates more than traces, the sulphates may be estimated directly by precipitation with barium chloride, or indirectly by evaporating and igniting an aliquot part of the filtrate from the tin (precipitated by ammonium nitrate), the result is then given as "metals not tin". A small quantity of iron must be allowed.

Metastannic Acid is obtained as the portion insoluble in excess of caustic soda solution.

Total Chlorine.—The estimation of the total chlorine is void of special interest, since the chlorides of the alkalis, etc., would have been found already amongst the "metals not tin". In the event of an estimation being required, the tin is first precipitated, and then the chlorine from the filtrate by means of silver nitrate, otherwise tin would be precipitated along with the silver chloride.

Requirements of Stannic Chloride.—It must contain as much pure $Sn\,Cl_4$ as possible, *i.e.*, contain chlorine equivalent to the tin, not too much free acid (up to 0·5 per cent.) and not too much basic salt (deficiency of chlorine not more than 1 per cent.) and tin oxychloride; further, as little as possible of metastannic acid, nitric acid, free chlorine, sulphates, salts of iron and the alkalis. Stannic chloride is now made on the large scale of a quality to satisfy every requirement, and technical inconveniences now occur more rarely than before its introduction.

Applications.—Formerly occurred in commerce as "pink salt," a double compound of stannic and ammonium chlorides, now as pure stannic chloride. For brightening (60 grammes soap, 3 grammes stannic chloride, 5 grammes crystallised soda, 20 grammes olive oil, boil well for ten minutes, and make up to 1,000 grammes; if the mixture acquires a brown colour it is useless); in wool-dyeing (alizarine with or without alum); as a cotton mordant (soap 5 to 10 : 1,000; $Sn.Cl_4$ of 4° B., aluminium acetate of 7° B. or sodium stannate with neutralised alum); in dyeing with ponceaux; in dyeing wood red (however makes the fibre hard); in particular as the silk weighting material *par excellence* (27 to 30° B.), this application has acquired great importance during the last twelve years.

Percentage of Tin and Specific Gravity.

Specific Gravity °B.	Tin per cent.	Specific Gravity °B.	Tin per cent.	Specific Gravity °B.	Tin per cent.
3	1	19	8·0	35	15·4
5	2	21	8·7	37	16·0
7	3	23	9·5	38	16·3
9	3·7	25	10·7	39	16·7
11	4·6	27	11·5	40	17·2
13	5·3	29	12·5	50	21·6
15	6·2	31	13·4	55	24·0
17	7·3	33	14·5	60	26·0

"**Tin Spirits**" contain stannous and stannic chlorides, with or without free hydrochloric and sulphuric acids, and vary in composition to an extraordinary degree. The chlorides of tin are estimated by the methods already described; the free acids are also to be determined.

In addition to stannous and stannic chlorides, a number of other preparations of tin come into the market, in which nitric and sulphuric acids, in addition to hydrochloric acid, have been used as solvents. Either stannous or stannic chloride may predominate in these solutions. The nitric acid may be estimated by titration with indigo solution (see *Water*), in which case the tin should first be precipitated. As a rule, these preparations are falling into disuse; they are being replaced by the pure compounds or by mixtures of these made by the consumer. The following are the formulæ for certain solutions of this type: "Nitro-chloride of tin," "Physic bath" or "Composition" = 5 kilos. of nitric acid + 5 kilos. of stannous chloride; "Scarlet Composition" = 5 kilos. of tin + 6 kilos. of nitric acid + 18 kilos. of hydrochloric acid + 6 litres of water; "Nitrate of tin" = 5 kilos. of tin + 20 kilos. of hydrochloric acid + 10 kilos. of nitric acid; "Sulphate of tin" = 5 kilos. of tin + 9 kilos. of hydrochloric acid + 2 kilos. of sulphuric acid + 2 litres of water; etc.

Application restricted, otherwise as given under *Stannous* and *Stannic Chlorides*, but not used for weighting silk.

Chromium Chloride, $Cr_2Cl_6 = 317{\cdot}3$. Commercially as a solution of 30° B., etc.

The percentage of chromium gives the value of the substance. It is estimated by precipitation and ignition to Cr_2O_3. When much iron is present, which at times is the case, a determination should be made (Fresenius, *Quant. Anal.*, i., 442). The solution should not be strongly acid, but should not be basic or contain much sulphuric acid.

Application.—As on mordant on cotton and silk, for adjectival colouring matters and especially for alizarine. It deposits the chromic oxide more readily upon the fibre than, *e.g.*, chrome alum (which see); also in calico-printing in order to produce the lake of the colouring matter.

FLUORIDES AND BIFLUORIDES.

Recently fluorides have come into use in the textile industries alongside the chlorides, though such use is at present partially experimental. Free hydrofluoric acid is not employed, although it is stated, in dilute solution, to impart a greater lustre to silk.

Alkaline Bifluorides, $KF.\ HF$ and $NaF.\ HF$. These should not contain too much foreign acid, much iron or other metals.

Application.—Used principally to replace cream of tartar and potassium bichromate. Up to the present no particularly favourable or unfavourable results have been recorded; the fabric, however, is made appreciably (1 to 2 per cent.) stronger.

Chromium Fluoride, $Cr_2F_6.\ 8H_2O = 362{\cdot}6$. This salt should contain about 42 to 43 per cent. of chromium oxide, should not be too acid and should be as far as possible free from iron, which metal may be estimated as in chromium chloride.

Application. — In calico-printing instead of chromium acetate, with which, however, it cannot compete; in mordanting wool instead of potassium bichromate, which is the chief application. The chromium fluoride black so obtained is said not to turn green, whilst potassium bichromate black fades towards green. It should, however, not be used in copper vessels, since copper fluoride is formed, which weakens the fibre. Wooden vessels with lead coils must be used, or, according to Kertész, if zinc strips be hung in copper vessels, the copper remains unattacked.

Chromium Oxyfluoride, or basic fluoride = chromium fluoride + chromium oxide. The percentage of chromium and the basicity are to be estimated as for ferric sulphate (which see).

Application.—As chromium fluoride.

Copper Fluoride has been recommended as an oxidising agent for aniline black, and for the after-oxidation; hitherto it has been little used.

Antimony Fluoride and **Aniline Hydrofluoride** have been recommended, but never introduced. The latter is especially recommended for aniline black on silk.

Antimony Fluoride Double Salts.—Antimony salt, $SbF_3.(NH_4)_2SO_4$, contains 47 per cent. of Sb_2O_3. Double antimony fluoride, $SbF_3.NaF$, contain 66 per cent. of Sb_2O_3. These compounds are offered as substitutes for tartar emetic. Before use a trace of soda should be added, in order to accelerate the formation of the lake (the colour then, however, rubs off more readily).

In printing, 2 to 5 grammes of antimony salt are added per kilo. of colour, and soda is added to the extent of $\frac{1}{5}$ to $\frac{1}{6}$ of the weight of the antimony salt.

Sulphuric Acid.

$H_2SO_4 = 98$; soluble in water in every proportion.

The strength is estimated from the specific gravity, or volumetrically, as with hydrochloric acid: 25 grammes are diluted to 1,000 c.c., 50 c.c. are taken and titrated with normal caustic soda; 1 c.c. of normal soda = 0·049 gramme of H_2SO_4 = 0·04 gramme of SO_3. In the case of very strong acid, above 96 per cent., the specific gravity cannot be used (see table), volumetric methods must then be employed, which is, indeed, always desirable when accuracy is required. In addition to the strength, the following impurities may be tested for: sodium sulphate (residue on ignition), gypsum, alumina, iron, lead, arsenic, zinc, copper, hydrochloric, nitric and sulphurous acids. The residue on ignition should not exceed tenths of 1 per cent. Sulphurous acid is detected by means of iodine (see *Fuming Sulphuric Acid* and *Sulphites*).

Specific Gravity of Sulphuric Acid at 15° C. (Kolb).

°B.	Specific Gravity.	100 parts by weight contain				1 litre contains kilos. of			
		SO_3 per cent.	H_2SO_4 per cent.	Acid of 60° B. per cent.	Acid of 53° B. per cent.	SO_3	H_2SO_4	Acid of 60° B.	Acid of 53° B.
0	1·000	0·7	0·9	1·2	1·3	0·007	0·009	0·012	0·013
1	1·007	1·5	1·9	2·4	2·8	0·015	0·019	0·024	0·028
2	1·014	2·3	2·8	3·6	4·2	0·023	0·028	0·036	0·042
3	1·022	3·1	3·8	4·9	5·7	0·032	0·039	0·050	0·058
4	1·029	3·9	4·8	6·1	7·2	0·040	0·049	0·063	0·074
5	1·037	4·7	5·8	7·4	8·7	0·049	0·060	0·077	0·090
6	1·045	5·6	6·8	8·7	10·2	0·059	0·071	0·091	0·107
7	1·052	6·4	7·8	10·0	11·7	0·067	0·082	0·105	0·123
8	1·060	7·2	8·8	11·3	13·1	0·076	0·093	0·120	0·139
9	1·067	8·0	9·8	12·6	14·6	0·085	0·105	0·134	0·156
10	1·075	8·8	10·8	13·8	16·1	0·095	0·116	0·148	0·173
11	1·083	9·7	11·9	15·2	17·8	0·105	0·129	0·165	0·193
12	1·091	10·6	13·0	16·7	19·4	0·116	0·142	0·182	0·211
13	1·100	11·5	14·1	18·1	21·0	0·126	0·155	0·199	0·231
14	1·108	12·4	15·2	19·5	22·7	0·137	0·168	0·216	0·251
15	1·116	13·2	16·2	20·7	24·2	0·147	0·181	0·231	0·270
16	1·125	14·1	17·3	22·2	25·8	0·159	0·195	0·250	0·290
17	1·134	15·1	18·5	23·7	27·6	0·172	0·210	0·269	0·313
18	1·142	16·0	19·6	25·1	29·2	0·183	0·224	0·287	0·333
19	1·152	17·0	20·8	26·6	31·0	0·196	0·233	0·306	0·357
20	1·162	18·0	22·2	28·4	33·1	0·209	0·258	0·330	0·385
21	1·171	19·0	23·3	29·8	34·8	0·222	0·273	0·349	0·407
22	1·180	20·0	24·5	31·4	36·6	0·236	0·289	0·370	0·432
23	1·190	21·1	25·8	33·0	38·5	0·251	0·307	0·393	0·458
24	1·200	22·1	27·1	34·7	40·5	0·265	0·325	0·416	0·486
25	1·210	23·2	28·4	36·4	42·4	0·281	0·344	0·440	0·513
26	1·220	24·2	29·6	37·9	44·2	0·295	0·361	0·465	0·539
27	1·231	25·3	31·0	39·7	46·3	0·311	0·382	0·489	0·570
98	1·241	26·3	32·2	41·2	48·1	0·326	0·400	0·511	0·597
29	1·252	27·3	33·4	42·8	49·9	0·342	0·418	0·536	0·625
30	1·263	28·3	34·7	44·4	51·8	0·357	0·438	0·561	0·654
31	1·274	29·4	36·0	46·1	53·7	0·374	0·459	0·587	0·684
32	1·285	30·5	37·4	47·9	55·8	0·392	0·481	0·616	0·717
33	1·297	31·7	38·8	49·7	57·9	0·411	0·503	0·645	0·751
34	1·308	32·8	40·2	51·1	60·0	0·429	0·526	0·674	0·785
35	1·320	33·8	41·6	53·3	62·2	0·447	0·549	0·704	0·820
36	1·332	35·1	43·0	55·1	64·2	0·468	0·573	0·734	0·856
37	1·345	36·2	44·4	56·9	66·3	0·487	0·597	0·765	0·892
38	1·357	37·2	45·5	58·3	67·9	0·505	0·617	0·791	0·921
39	1·370	38·3	46·9	60·0	70·0	0·525	0·642	0·822	0·959
40	1·383	39·5	48·3	61·9	72·1	0·546	0·668	0·856	0·997
41	1·397	40·7	49·8	63·8	74·3	0·569	0·696	0·891	1·038
42	1·410	41·8	51·2	65·6	76·4	0·589	0·722	0·925	1·077

Specific Gravity of Sulphuric Acid at 15° *C.* (*Kolb*).—contd.

°B.	Specific gravity.	100 parts by weight contain				1 litre contains kilos. of			
		SO_3 per cent.	H_2SO_4 per cent.	Acid of 60° B. per cent.	Acid of 53° B. per cent.	SO_3	H_2SO_4	Acid of 60° B.	Acid of 53° B.
43	1·424	42·9	52·8	67·4	78·5	0·611	0·749	0·960	1·108
44	1·438	44·1	54·0	69·1	80·6	0·634	0·777	0·994	1·159
45	1·453	45·2	55·4	70·9	82·7	0·657	0·805	1·030	1·202
46	1·468	46·4	56·9	72·9	84·9	0·681	0·835	1·070	1·246
47	1·483	47·6	58·3	74·7	87·0	0·706	0·864	1·108	1·290
48	1·498	48·7	59·6	76·3	89·0	0·730	0·893	1·143	1·330
49	1·514	49·8	61·0	78·1	91·0	0·754	0·923	1·182	1·378
50	1·530	51·0	62·5	80·0	93·3	0·780	0·956	1·224	1·427
51	1·540	52·2	64·0	82·0	95·5	0·807	0·990	1·268	1·477
52	1·563	53·5	65·5	83·9	97·8	0·836	1·024	1·311	1·529
53	1·580	54·9	67·0	85·8	100·0	0·867	1·059	1·355	1·580
54	1·597	56·0	68·6	87·8	102·4	0·894	1·095	1·402	1·636
55	1·615	57·1	70·0	89·6	104·5	0·922	1·131	1·447	1·688
56	1·634	58·4	71·6	91·7	106·9	0·954	1·170	1·499	1·747
57	1·652	59·7	73·2	93·7	109·2	0·986	1·210	1·548	1·804
58	1·672	61·0	74·7	95·7	111·5	1·019	1·248	1·599	1·863
59	1·691	62·4	76·4	97·8	114·0	1·055	1·292	1·654	1·928
60	1·711	63·8	78·1	100·0	116·6	1·092	1·336	1·711	1·995
61	1·732	65·2	79·0	102·3	119·2	1·129	1·384	1·772	2·065
62	1·753	66·7	81·7	104·6	121·9	1·169	1·432	1·838	2·137
63	1·774	68·7	84·1	107·7	125·5	1·219	1·492	1·911	2·226
64	1·796	70·6	86·5	110·8	129·1	1·268	1·554	1·990	2·319
65	1·819	73·2	89·7	114·8	138·8	1·332	1·632	2·088	2·434
66	1·842	81·6	100·0	128·0	149·3	1·503	1·842	2·358	2·750

The determinations of Kolb are unreliable for sulphuric acid containing more than 90 per cent. of H_2SO_4. The results of Lunge and Naef are given in the table on the opposite page, the values marked * have been directly observed, the others are obtained by interpolation. These figures relate to *chemically pure* acid; the specific gravities of very concentrated commercial acid are higher.

Application.—Used on a very large scale. In dyeing wool in the acid bath, silk in acidified "boiled-off liquor" (certain dyes, such as eosine, cannot withstand sulphuric acid, in their case acetic acid must be used); in stripping the colour-lake from the fibre; in carbonising loose wool (2° to 5°B.);

in calico bleaching instead of hydrochloric acid, which often contains iron and free chlorine; in discharging indigo; in calico-printing to remove resists; in the preparation of Turkey red oil (sulpholeate); together with sodium sulphate ($NaHSO_4$) as a substitute for tartar; in the preparation of indigo carmine (fuming sulphuric acid); in mordanting wool with bichromate and sulphuric acid; for cleaning copper pans, etc., etc.

Specific Gravity of Highly Concentrated Sulphuric Acid (Lunge and Naef).

H_2SO_4 per cent.	Specific gravity.	°B.	H_2SO_4 per cent.	Specific gravity.	°B.
90	1·8185	65·1	*95·97	1·8406	
*90·20	1·8195		96	1·8406	66·0
91	1·8241	65·4	97	1·8410	
*91·48	1·8271		*97·70	1·8413	
92	1·8294	65·6	98	1·8412	
*92·83	1·8334		*98·39	1·8406	
93	1·8339	65·8	*98·66	1·8409	
94	1·8372	65·9	99	1·8403	
*94·84	1·8387		*99·47	1·8395	
95	1·8390	66·0	*100·00	1·8384	

Fuming or Nordhausen Sulphuric Acid, H_2SO_4 + (varying quantities of) SO_3.

A solution of sulphuric anhydride in sulphuric acid. It is valued according to the proportion of free anhydride, which must be estimated in assessing the value. Sulphurous acid, which may be present, must also be estimated by means of standard iodine solution and deducted from the total acid. All the processes described in the literature for the evaluation of fuming sulphuric acid (Clar and Gaier, *Chem. Ind.*, iv., 251; Lunge and Hurter, *Alkali Makers' Pocket-book*, 1884, 100; Winkler, etc.), are not sufficiently quick and simple for practical use. The following method is easy and certain of execution, and, whilst requiring little time, gives accurate results. A dry ordinary thin-walled test-tube (about 18 mm. wide) is drawn out, about 4 cms. from the bottom, to a capillary 3 to 4 cms. long. It is weighed, and the capillary dipped into the acid, which is best contained in a narrow-necked flask, and should have been thoroughly mixed. The upper part of the tube is then heated by means of a Bunsen burner until so much air has been expelled that 8 to 10 grammes of the acid enter the tube on cooling. The capillary is at once fused together, the glass cleaned, again weighed and brought into a thick-walled, stoppered litre flask containing 200 to 300 c.c. of water. The test tube is then broken by shaking the flask, cooling at the same time; the fumes of anhydride are absorbed by the water within a few minutes. When this has occurred and the contents have cooled, they are separated from the broken glass by running through a funnel, made up to 1,000 c.c. and 250 c.c. titrated with normal caustic soda.

Example: 9·7104 grammes of fuming sulphuric acid taken; the 250 c.c. required 52·38 c.c. of normal soda; 52·38 × 0·04 × 4 = 8·3808 grammes of SO_3 in 9·7104 grammes of acid = 86·3 per cent. of SO_3; 100 − 86·3 = 13·7 per cent. of water, which is united to SO_3; 13·7 of water correspond to 13·7 × 4·44 = 60·82 per cent. of SO_3 combined with the water. Thus 86·3 − 60·82 = 25·48 per cent. of SO_3 is present in the anhydrous condition. If, further, we were to find, by titration of the sulphurous acid with iodine solution, 0·16 per cent. of SO_2, then, since 0·16 grammes of SO_2 = 0·20 grammes of SO_3, there would be present 25·48 − 0·20 = 25·28 per cent. of SO_3 as pure anhydride.

Application.—To dissolve indigo in preparing indigo carmine; in making Turkey red oils and olive oil emulsions for Turkey red dyeing.

SULPHATES.

Sulphates are frequently adulterated, or contaminated, by chlorides, sand, free acids, carbonates, iron and other metals. As in the case of the chlorides, the general requirements are dryness, complete solubility and neutrality. The more important sulphates are mentioned individually.

Sodium Sulphate, Glauber's Salt, $Na_2SO_4 . 10H_2O = 322$; solubility, cold water = 5 : 100; hot water = 42·5 : 100.

Sodium chloride is a frequent impurity.

Application.—In wool-dyeing, generally together with sulphuric acid, or as the technical bisulphate ("Tartar substitute"). It causes the fibre to take up the dye more evenly.

Sodium Bisulphate, Tartar Substitute, $NaHSO_4 . H_2O = 138$; very soluble in water.

The acidity may be estimated by titration with normal caustic soda, 1 c.c. = 0·138 gramme of $NaHSO_4 . H_2O$.

Application.—As sodium sulphate, generally only in wool-dyeing, more rarely in silk-dyeing.

Calcium Sulphate, Gypsum, $CaSO_4 . 2H_2O = 172$; solubility, cold water = 1 : 490; hot water = 1 : 460.

Frequently contains carbonate, alkaline sulphate and chloride.

Application.—In dressing and weighting fabrics.

Magnesium Sulphate, Epsom Salts, $MgSO_4 . 7H_2O = 246$; solubility, cold water = 26 : 100; hot water = 71·5 : 100.

Often contains chlorides and foreign (alkali) metals.

Application.—Almost exclusively in finishing; without injury a quantity 4 to 6 times greater than the permissible amount of magnesium chloride may be added. It makes the fabric thicker, moister and softer.

This salt is also added, as an adulterant, to tin crystals and tannin (which see).

Lead Sulphate, $PbSO_4 = 302$; solubility, cold water = 1 : 22,800; hot water very slight.

Comes into commerce in paste form, and is often adulterated by barium sulphate and gypsum.

Application.—In printing blues.

Ferrous Sulphate, Green Vitriol, Copperas, $FeSO_4 . 7H_2O = 278$; solubility, cold water = 60 : 100; hot water = 333 : 100; 25·9 per cent. of FeO.

Commercial ferrous sulphate is generally fairly pure. As impurities it contains more or less ferric oxide, lime, chlorine, free sulphuric acid, occasionally also copper, zinc and alum. Copper is especially harmful to the alizarine dyes; zinc and alumina are also to be avoided. According to the intended use, a greater or less degree of oxidation is permissible and desired. Alumina is detected by precipitating the iron from a boiling solution with caustic soda, after previous oxidation with nitric acid or bromine water. The alumina remains dissolved and is precipitated as hydrate from the filtrate on adding excess of ammonium chloride (or hydrochloric acid to acid reaction and then ammonia to alkaline reaction).

The value is essentially determined by the proportion of total and ferrous iron.

Total iron is estimated gravimetrically by precipitating the oxidised solution with ammonia, filtering and igniting to Fe_2O_3, or volumetrically as described in detail under *Ferric Sulphate* (which see).

Ferrous Oxide.—(*a*) The ferrous oxide is estimated by direct titration with permanganate: 50 grammes are dissolved to 1,000 c.c.; to 50 c.c. of the solution are added 20 c.c. of sulphuric acid (1 : 3) and the mixture then titrated with $\frac{1}{5}$ N permanganate, 1 c.c. of which = 0·0144 gramme of FeO = 0·0566 gramme of $FeSO_4 . 7H_2O$.

(*b*) Bichromate solution may be used instead of permanganate, the process is then the reverse of the titration of chromic acid by ferrous salt (see *Potassium Bichromate*), 1 c.c. of $\frac{1}{10}$ N potassium bichromate = 0·0072 gramme of FeO or 0·0278 gramme of $FeSO_4 . 7H_2O$.

The difference between the total iron and ferrous iron corresponds to the ferric oxide, 1 c.c. of $\frac{1}{5}$ N permanganate = 0·016 gramme of Fe_2O_3.

Applications.—Very extensive. In indigo-dyeing (1 part of indigo, 3·5 to 4 parts of ferrous sulphate, 4 parts of lime); in dyeing cotton black, in alizarine-dyeing, in dyeing wool black, for iron chamois. Ferrous sulphate often comes into the market mixed with copper sulphate.

Aluminium Sulphate, Sulphate of Alumina, $Al_2(SO_4)_3 . 18H_2O$ = 664·8; solubility, cold water = 85 : 100; hot water = 1,130 : 100.

As impurities are found: iron, zinc, copper, lead, chromium, alkalis, chlorine, free sulphuric acid.

Alumina may be estimated by dissolving 25 grammes to 1,000 c.c., and heating 50 c.c. of the solution with ammonia and ammonium chloride at a temperature near the boiling point, until the ammonia is nearly all driven off. The solution must, however, remain distinctly alkaline. The precipitate is filtered off, dried, ignited, weighed and calculated as Al_2O_3.

In the precipitate *iron* may be estimated colorimetrically and deducted from the alumina.

The *basicity* may be estimated as in ferric sulphate (which see).

Free sulphuric acid is detected by tropaeolin OO. Watson Smith (*Journ. Soc. Dyers and Col.*, 1884, 35) recommends for this purpose ferric acetate, which loses its red colour in the presence of traces of mineral acids. The free acid can be approximately estimated by titration of an alcoholic extract of the sulphate with $\frac{1}{10}$ N alkali and phenolphthaleïn.

The most accurate method is that of Beilstein and Grosset (*Zeits. anal. Chem.*, 1890, 77): 1 to 2 grammes of the sulphate are dissolved in 5 c.c. of water, 15 c.c. of a cold saturated neutral solution of ammonium sulphate added, and the mixture stirred for fifteen minutes; 50 c.c. of 95 per cent. alcohol are then added, when the whole of the aluminium sulphate is precipitated as ammonia alum, whilst the free sulphuric acid remains in solution. The liquid is filtered, the whole or an aliquot part evaporated on the water-bath, the residue taken up in water and titrated with $\frac{1}{10}$ N caustic soda and phenolphthaleïn.

Iron can be detected and estimated colorimetrically with potassium ferro- or ferricyanide, tannin, potassium sulphocyanide, etc. H. v. Kéler has carried out an exhaustive research on the permissible amount of iron in aluminium sulphate. The results were as follows: aluminium sulphate for Turkey red dyeing should not contain more than 0·001 per cent. of total iron, a larger quantity is harmful; ferric salts are more harmful than ferrous. In calico-printing the iron may rise to 0·00524 per cent., more may be harmful. Zinc salts are always harmful.

Application.—Very extensive as a mordant in dyeing and printing, as a waterproofing material, etc. For cotton generally used as basic salt (1 kilo. of normal salt + 160 grammes of calcined soda), followed by fixing with ammonia, ammonium carbonate, sodium phosphate, arsenate or silicate, soap, Turkey red oil; for wool, cold to hot, as normal salt with tartar (4 : 3); for silk the normal and basic salt, the fabric remains in the cold solution twelve to twenty-four hours (also see *Alum*).

Ferric Sulphate, Nitrate of Iron, Iron Mordant, $Fe_2(OH)_2(SO_4)_2$ to $Fe_4(OH)_2(SO_4)_5$.

Ferric sulphate is the best for most purposes when its composition corresponds to the formula $Fe_8(OH)_6(SO_4)_9$, *i.e.*, when $SO_3 : Fe_2O_3 = 1 : 1·125$. As a rule it is more acid. It is advisable to take care that the basicity is always, as far as possible, the same, since the basicity is of the greatest importance to the mordant.

The following are characteristics of commercial preparations:—

	Fe_2O_3 per cent.	SO_3 per cent.	FeO per cent.	Cl, N_2O_5	Ratio $SO_3 : Fe_2O_3$
I.	18·4	22·0	0·4	trace	1 : 1·2
II.	19·4	23·3	0·25	—	1 : 1·2
III.	21·7	25·0	0·30	—	1 : 1·15
IV.	21·16	24·23	0·35	—	1 : 1·14

If the mordant is too basic, the fabric is injured and the mordant readily separates precipitates: if it is too acid, too little ferric oxide is fixed. However, the requirements of consumers vary from time to time.

A complete examination would be similar to that of stannic chloride; the following would be determined: specific gravity (picnometer, hydrometer), ferrous oxide, total iron, ferric oxide, total acid, sulphuric acid, chlorine, nitric acid, alkalis, etc.

The *specific gravity* is taken by means of a 50 c.c. specific gravity bottle; this 50 c.c. is then diluted to 1,000 c.c.

Ferrous Oxide.—200 c.c. of this solution are acidified with sulphuric acid and titrated with $\frac{1}{5}$ N permanganate, 1 c.c. of which = 0·0144 gramme of FeO = 0·0112 gramme of Fe.

Total Iron and thence *Ferric Oxide.*—(*a*) 20 c.c. of the dilute solution are reduced with about 20 grammes of zinc (granulated or in rods) and an excess of sulphuric acid, until the solution no longer gives a reaction with potassium sulphocyanide. The reduction is performed in a small flask with a Bunsen valve. The solution is then poured off from the

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undissolved zinc, sulphuric acid added, and titrated with $\frac{1}{5}$ N permanganate. The undissolved zinc is weighed and allowance made for the consumption of permanganate by the dissolved zinc (this correction is determined once for all for the particular sample of zinc, by titrating a solution of 50 to 100 grammes in sulphuric acid). The difference between total iron and ferrous iron gives the ferric oxide, *e.g.*, 7 grammes of zinc, when dissolved in sulphuric acid, require 0·1 c.c. of $\frac{1}{5}$ N permanganate; 14 grammes of zinc were used (= 0·2 cc. of permanganate), and in the titration 20·55 c.c. of permanganate were required, or deducting the 0·2 c.c. due to the zinc, 20·35 c.c. Therefore in 1 c.c. of the solution there is 20·35 × 0·016 = 0·3356 gramme of Fe_2O_3. The specific gravity of the solution was found to be 1·53834, thence 1·53834 : 0·3556 = 100 : x, or x = 21·16 per cent. of Fe_2O_3. If the ferrous oxide previously found be deducted, the result gives the iron present as ferric oxide.

(*b*) The total iron may also be estimated gravimetrically by precipitating 20 c.c. of the solution with ammonia. It is then heated near the boiling point until the smell of ammonia becomes slight, the precipitate filtered off, ignited and weighed as Fe_2O_3. If ferrous salt be present, which is generally the case, before precipitation the solution is oxidised with 2 to 3 drops of dilute nitric acid.

Total acid.—(*a*) 100 c.c. of the solution are mixed in a 500 c.c. flask with 60 or 70 c.c. of normal soda, the mixture boiled, allowed to cool, made up to 500 c.c. and an aliquot part (250 c.c.) filtered. The excess of soda is now estimated by titration with normal sulphuric acid and methyl orange. From the excess the amount used is calculated; 1 c.c. of normal soda = 0·040 gramme of SO_3.

Example: 100 c.c. of solution + 60 c.c. of normal soda made up to 500 c.c.; 200 c.c. require 4·525 c.c. of normal sulphuric acid; 200 : 4·525 = 500 : x; x = 11·31, 60 − 11·31

= 48·69 c.c. of normal soda used; 48·69 × 0·04 = 1·9476 grammes of SO_3; 100 c.c. of solution correspond to 7·7906 grammes of mordant; 7·7906 : 1·9476 = 100 : x, thence x = 25·00 per cent. of SO_3.

(*b*) The following process for the estimation of the total acid is simpler: 50 c.c. of the solution are decomposed with much hot water, heated for some time until the decomposition is complete (on the water-bath or over the naked flame to near the boiling point), and this solution, without filtering, titrated directly with normal soda, using phenolphthaleïn as indicator. Direct titration is permissible here, which is not the case with the chlorides of tin, because excess of soda has no action upon the ferric hydroxide. The method is accurate. In approaching the end of the titration, the liquid is allowed to stand for a moment from time to time, in order that it may be seen, from the supernatant solution, whether the acid is neutralised or not, *i.e.*, whether the liquid is rose-coloured or still colourless. 1 c.c. of normal soda = 0·04 gramme of SO_3.

Sulphuric Acid.—If it is required to estimate the sulphuric acid as such, and not merely the total acid, it is precipitated in 10 c.c. of the solution by barium chloride, and the barium sulphate weighed in the ordinary manner. The weight of barium sulphate multiplied by 0·34334 gives the SO_3. The difference between the total acid and sulphuric acid is to be ascribed to foreign acids—hydrochloric, nitric, sulphurous. The smaller the quantity of these foreign acids, the better. In the case of a good mordant the difference should not materially exceed 1 per cent.

Hydrochloric acid may also be estimated in the ordinary manner by means of silver, but this is seldom required.

Nitric acid, which is almost always present in appreciable quantity, may be titrated by means of indigo solution as in water, or estimated by the gasometric method.

Practical Test.—In the case of iron mordant a practical test is often very important; it may be carried out with wool or silk. For example, several skeins of cotton each of 10 grammes weight are well washed and stirred in a decoction of tannin, sumach, etc., for one hour, taken out, unwound and immersed in dilute solutions of the sample (5 c.c. in 250 c.c. of water) or of a standard sample of known quality. After twenty to thirty minutes they are taken out, washed, dried and compared. They may also be dyed with logwood. Silk is weighed before and after the experiment, in order to estimate the quantity of ferric oxide fixed. The more iron is fixed on the fibre, the better is the mordant, other things being equal. The weighed silk (the largest convenient amount) is mordanted for one hour in a solution of 30° B., well washed, treated with water at 50° C., then with Marseilles soap at the boil, washed, dried at the ordinary temperature and weighed.

Application.—Commercial ferric sulphate is generally a solution of 50° to 60° B.; it is principally used as a solution of 30° B. in black silk-dyeing as weighting and bottoming for Prussian blue; it is used for similar purposes in black cotton-dyeing, in printing the so-called "dyers' black," which has in recent years been largely supplanted by aniline black.

Copper Sulphate, Blue Vitriol, Bluestone, $CuSO_4 . 5H_2O = 249 \cdot 5$; solubility, cold water = 40 : 100; hot water = 203 : 100; 25 per cent. Cu.

The value is according to the percentage of pure copper sulphate; the salt should also give a clear solution in water, and not contain too much iron, lime and alkali salts (or zinc).

25 grammes are dissolved to 1,000 c.c.

Sulphuric Acid.—25 c.c. of this solution are acidified with hydrochloric acid and precipitated with barium chloride, the barium sulphate is weighed and calculated to sulphuric acid.

Copper.—25 c.c. are acidified with sulphuric acid and precipitated with sulphuretted hydrogen :—

(*a*) The copper sulphide is either ignited in a Rose crucible with fine precipitated sulphur in a current of hydrogen to constant weight; or

(*b*) the sulphide is roasted, moistened with nitric acid and ignited to constant weight, by which process it is quantitatively converted into copper oxide, CuO; or

(*c*) 25 c.c. of the solution are precipitated with caustic soda, the liquid boiled for some time, filtered after well washing by decantation, the precipitate well washed on the filter until the filtrate is neutral, then dried, ignited and weighed as CuO; 79·3 CuO = 79·3 Cu_2S = 63·3 Cu = 249·3 $CuSO_4 . 5H_2O$;

(*d*) or the copper is estimated by one of the many volumetric processes (see Fresenius, *Quant. Anal.*, i. 258). The Volhard-Henriques method, though little used, must be regarded as the best, since it is rapid and easy of executions, very accurate and requires no special solutions. 30 grammes of copper sulphate are dissolved to 500 c.c., 5 c.c. of the solution are mixed with excess of sulphurous acid in a 100 c.c. flask and 20 c.c. of $\frac{1}{10}$ N ammonium sulphocyanide added, the flask is filled up to the mark, well shaken and the liquid filtered; 50 c.c. of the filtrate are mixed with 10 c.c. (excess) of $\frac{1}{10}$ N silver nitrate solution, acidified with nitric acid, iron alum added as indicator, and the excess of silver titrated with $\frac{1}{10}$ N ammonium sulphocyanide, until the red coloration appears.

Calculation: Suppose that 5·6 c.c. of $\frac{1}{10}$ N sulphocyanide solution are used in titrating the excess of silver, then in 2·5 c.c. of the original solution there is 5·6 × 0·00794 gramme of Cu = 5·6 × 0·012475 gramme of $CuSO_4 . 5H_2O$, from which the percentage may be calculated.

Iron.—200 c.c. of the solution are oxidised with two drops of nitric acid and precipitated by excess of ammonia. The copper goes into solution, the iron is precipitated, it is filtered off, or redissolved and again precipitated, ignited and weighed as Fe_2O_3, or estimated colorimetrically.

Application.—Used as an oxidising agent for aniline black (copper sulphide is also used, it is prepared from the sulphate); in dyeing browns with catechu (25 parts of copper sulphate to 100 parts of catechu); in dyeing a blue from acid green + acid violet ($\frac{1}{3}$ $CuSO_2$ + $\frac{2}{3}$ alum); for "Jäger" green; in mordanting wool for logwood blacks; for an after-treatment of many direct-dyeing colouring matters, such as benzoazurine, in order to increase the fastness to light; for "chrome" black (0·5 per cent. $K_2Cr_2O_7$ + 1 per cent. $CuSO_4 . 5H_2O$, or 2 per cent. $K_2Cr_2O_7$ + 1·5 per cent. $CuSO_4 . 5H_2O$), etc.

ALUMS.

Potash alum, $K_2SO_4 . Al_2(SO_4)_3 . 24H_2O = 948$; solubility, cold water = 9·5 : 100; hot water = 357 : 100.

Soda alum, $Na_2SO_4 . Al_2(SO_4)_3 . 24H_2O = 917$; solubility, cold water = 110 : 100; hot water, very soluble.

Ammonia alum, $(NH_4)_2SO_4 . Al_2(SO_4)_3 . 24H_2O = 904·4$; solubility, cold water = 9 : 100; hot water = 422 : 100.

Iron alum, $Fe_2(SO_4)_3 . K_2SO_4 . 24H_2O = 1006·5$; solubility, cold water = 20 : 100; hot water, very soluble.

Chrome alum, $Cr_2(SO_4)_3 . K_2SO_4 . 24H_2O = 999$; solubility, cold water = 20 : 100; hot water = 50 : 100; 13·5 per cent. of Cr_2O_3.

The alums are extensively used in the textile industries. The commercial article is generally very pure. In an examination the following points are to be considered: In the case of potash and soda alums the percentage of alumina is of the first importance; the percentage of water may also be taken, and free acid (as with aluminium sulphate) and iron tested for. The same may be said of ammonia alum. In the case of iron alum iron, in chrome alum chromium occupies the place of aluminium ; the proportion of these two metals indicates the purity of the compound. Iron alum should also contain no ferrous sulphate, or very little (detected and estimated as in ferric sulphate). Chrome alum, which is generally used in the state of the basic sulphate $Cr_2(SO_4)_2(OH)_2$, frequently contains tarry matter, gypsum and sodium sulphate, all of which should, as far as possible, be absent. The chromium is estimated by precipitation with ammonia. If the chrome alum gives an intense iron reaction, the chromium must be separated from the iron. In other respects a detailed examination is to be conducted as with the corresponding sulphates.

Application.—Very extensive. In dyeing with certain dyestuffs such as Victoria Blue (3 to 5 per cent. of alum + acetic acid) etc. ; as a mordant in alizarine-dyeing ; in wool-dyeing as a mordant (7 to 10 per cent. of alum + 1 per cent. of tin crystals), almost entirely replaced by potassium bichromate ; in cochineal-dyeing (10 to 12 per cent. of alum + 2 to 3 per cent. of tartar) ; in dyeing flannel with ponceaux, replacing cochineal (0·5 to 1 per cent. of alum + ponceau + trace of stannic chloride) ; in dyeing eosine on wool (alum gives a very bright eosine lake) ; with Water Blue ; in Turkey red dyeing (for this purpose alum must be free from iron or at most contain 0·001 per cent. ; solutions of 1° B. to 5° to 8° B. are used as mordants) ; with indoïn blue ; in printing on wool (alizarine orange and red).

Iron alum is little used : as a mordant for wool blacks, occasionally also for alizarine.

Chrome alum is also rarely used. It is principally important as forming the starting point for the preparation of other chromium salts, *e.g.* chromium acetate, chloride, etc.

It has also been recommended for mordanting wool, but it has not been introduced, since it does not precipitate the chromium oxide quite evenly on the fibre, and cannot in this property equal potassium bichromate.

Nitric Acid and Nitrates.

Nitric Acid.—$HNO_3 = 63$. When no foreign acid is present, the strength may be estimated by means of the specific gravity, or volumetrically. 50 grammes are diluted to 1,000 c.c. and 50 c.c. of the solution titrated with normal caustic soda, 1 c.c. of which = 0·063 gramme of HNO_3.

In testing for impurities, chlorine, sulphuric acid, metals, iron, alkali salts and nitrous acid may be expected. If the acid is contaminated by other acids and the proportion of pure nitric acid is to be determined, the nitric oxides may be estimated. However, nitric acid is of so little importance in the textile industries that it is not necessary to introduce this complicated gasometric method here.

Application.—Very restricted. In colouring nitrogenous materials (feathers, silk) which become yellow by reason of the nitro-compounds produced, but are at the same time considerably weakened (2 parts of HNO_3 + 1 part HCl diluted to 2° B.); in the production of imitation selvages on fabrics for export (nitric acid + gum + starch printed on, in order to give the appearance of yarn-dyed fabrics, which have yellow selvages); as a reagent for indigo, which does not now give a distinguishing reaction, since many other blues give the same spot, *e.g.* the blue obtained from acid green + acid violet; for engraving printing rollers.

Sodium Nitrate, Chili Saltpetre, $NaNO_3 = 85$; Solubility, cold water = 80 : 100; hot water = 200 : 100.

Nitric acid is estimated, as in water, by means of indigo solution. Chloride and sulphate occur as impurities.

Application.—Very restricted, occasionally used for the after-oxidation of aniline black.

Silver Nitrate, $AgNO_3 = 170$; solubility, cold water = 120 : 100; hot water = 1,000 : 100.

The percentage of silver is all-important. The salt is titrated with decinormal sodium chloride solution, using neutral potassium chromate as indicator, until the brown coloration just appears; 1 c.c. of decinormal NaCl solution = 0·017 gramme of $AgNO_3$.

Application.—Almost only for marking inks ($AgNO_3 + NH_3$ + gum, written or stamped on). The writing is not as sensitive to dilute alkalis and acids as to chlorine.

Lead Nitrate, $Pb(NO_3)_2 = 330$; solubility, cold water = 48·3 : 100; hot water = 139 : 100.

The percentage of lead indicates the value, it is precipitated as sulphate by means of sulphuric acid, filtered, ignited and weighed as $PbSO_4$.

Application.—For chrome yellow and orange; as the starting point for other lead compounds.

Ferrous Nitrate, $Fe(NO_3)_2 . 6H_2O = 288$; soluble in cold water, decomposed by hot water.

Examined in a similar manner to ferrous sulphate. Nitric acid estimated as in sodium nitrate.

Application.—Now rarely or never used; for rust yellow on cotton, Prussian blue on wool.

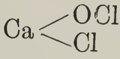
Ferric Nitrate, $Fe_2(NO_3)_6 . 18H_2O = 808$; readily soluble in hot and cold water.

Examined as ferric sulphate, except in regard to the nitric acid. To be tested for sulphuric acid.

Application.—As restricted as the last-mentioned compound; in black cotton-dyeing; in silk-dyeing it has been entirely replaced by basic ferric sulphate.

Rarer Nitrates.—For certain special purposes in printing the following nitrates are occasionally used; they are obtained by double decompositions: calcium, aluminium, magnesium, copper and chromium nitrates, the double chromium nitrate acetate.

Chlorine-Oxygen Compounds.

Bleaching Powder is a mixture, in varying proportions, of calcium hypochlorite, chloride and hydroxide with water. The active constituent is best expressed by the formula $CaCl_2O$ (according to Odling:  $\mathrm{Ca}<^{\mathrm{OCl}}_{\mathrm{Cl}}$).

The value is always determined by the "available chlorine". According to Schützenberger, however, the compound does not act by means of the chlorine, but by reason of the free oxygen produced by its decomposition ($CaCl_2O = CaCl_2 + O$). There are the following methods for the estimation of the chlorine:—

I. Gay Lussac's method. The bleaching powder is titrated in aqueous solution with sodium arsenite, using indigo as the indicator. This method has long been disused on account of its inaccuracy: in dilute solution arsenious acid no longer has any action.

II. Pennot's method with alkaline arsenic solution and potassium iodide starch paper as indicator.

III. Iodometric methods.

The two latter are most generally used; they will be briefly described.

II. (*a*) Pennot's solution: 4·425 grammes of pure arsenious acid and 13·0 grammes of pure crystallised soda are dissolved in 600 to 700 c.c. of warm water and the solution made up to 1,000 c.c. when cold; 1 c.c. of the solution corresponds to 0·004425 gramme of As_2O_3 = 1 c.c. of chlorine gas at 0° C. and 760 mm. pressure.

10 grammes of bleaching powder are well ground with water and the paste, together with the residue, filled up to

1,000 c.c.; 50 c.c. of the solution are titrated with the above Pennot's solution, until no blue coloration is produced on potassium iodide starch paper, which is the case when all the active chlorine has been used.

Example: 50 c.c. of bleaching powder solution required 40 c.c. of Pennot's solution, *i.e.*, 50 c.c. of bleaching powder solution correspond to 40 c.c. of chlorine gas, or 0·5 gramme of bleaching powder gives off 40 c.c. of chlorine, hence 1 kilo. of bleaching powder can evolve 80 litres of chlorine measured as 0° C. and 760 mm. pressure ($2CaCl_2O + As_2O_3 = 2CaCl_2 + As_2O_5$).

(*b*) In place of Pennot's solution, $\frac{1}{10}$ N arsenic solution (4·95 grammes of pure As_2O_3 dissolved by means of soda to 1,000 c.c.) may be used; 1 c.c. of $\frac{1}{10}$ N sodium arsenite solution = 0·00354 gramme of available chlorine.

III. Iodometric methods: 7·1 grammes of bleaching powder are ground with water and the mixture made up to 1,000 c.c.; 50 c.c. (= 0·354 gramme) are measured by means of a pipette into a beaker containing 1 litre of distilled water, 1 gramme of potassium iodide and about 10 drops of hydrochloric acid are then added. The liquid is stirred round slowly once, and $\frac{1}{10}$ N thiosulphate run in quickly from a burette, without stirring, until the colour is slightly yellow. Starch solution is then added and the titration continued slowly until the blue colour disappears. $Cl_2 + 2KI = 2KCl + I_2$; $I_2 + 2Na_2S_2O_3 = Na_2S_4O_6 + 2NaI$; 1 c.c. of $\frac{1}{10}$ N thiosulphate = 0·00354 gramme of chlorine. If, for example, 35·25 c.c. of $\frac{1}{10}$ N thiosulphate are used, then the percentage of available chlorine $= \frac{35{\cdot}25 \times 0{\cdot}00354 \times 100}{0{\cdot}354} = 35{\cdot}25$, *i.e.*, 1 c.c. of $\frac{1}{10}$ N thiosulphate = 1 per cent. of available chlorine.

IV. The method of Baumann (*Zeits. angew. Chem.*, 1890, 73) may also be mentioned; it is based upon adding hydrogen peroxide and titrating with potassium permanganate:

$CaCl_2O + H_2O_2 = CaCl_2 + H_2O + O_2$. The excess of hydrogen peroxide is titrated with permanganate: $2KMnO_4 + 5H_2O_2 + 3H_2SO_4 = K_2SO_4 + 2MnSO_4 + 8H_2O + 5O_2$.

As has already been seen in II. and III. the strength of bleaching powder may be expressed either in chlorometric degrees (II.), as is customary in France, or in percentages of available chlorine (III.), the usual custom in England and Germany. The chlorometric degrees give the number of litres of (available) chlorine gas (measured at 0° C. and 760 mm. pressure) contained in 1 kilo. of bleaching powder; the English and German degrees are percentages of available chlorine. The connection between the two systems is as follows:—

Chlorometric degrees multiplied by 0·3178 = percentage of available chlorine.

Percentage of available chlorine divided by 0·3178 = chlorometric degrees.

French Scale.	English and German Scale.	French Scale.	English and German Scale.
63	20·02	100	31·78
70	22·24	105	33·36
75	23·83	110	34·95
80	28·42	115	36·54
85	27·01	120	38·13
90	28·60	125	40·67
		(The best bleaching powder.)	

The question whether the deposit or insoluble residue should be included in titrations of bleaching powder is an important point upon which there should be an understanding. Fresenius titrates it together with the solution, other authorities on textile matters take the opposite course, since, as they very properly say, the insoluble matter is unutilised and unutilisable in practice, and in a technical analysis the technical conditions must be considered.

Chemically pure calcium hydrochlorite contains theoretically 48·9 per cent. of chlorine; in the manufacture of bleaching powder, however, a product containing more than 39 per cent. is not obtained.

Application.—Bleaching powder is the bleaching material *par excellence* for vegetable materials; at the present time solutions over 1° B. are rarely, if ever, used, whilst formerly they were used at 3° B. and over. At times a few drops of hydrochloric acid are added; acetic and formic acids have also been proposed, but the carbonic acid of the air is quite sufficient. A strong solution is used to produce cream-coloured linen. Wool and silk are turned yellow by chlorine, are rotted and altered in structure. Cotton is only altered in strong solutions, oxycellulose being formed and the fibre to a certain extent "animalised". In recent years wool has also been treated with chlorine in order to produce certain lustrous and contracted effects.

Alkaline Hypochlorites. Eau de Javelle, KOCl; Eau de Labarraque, NaOCl.

These compounds are only stable in solution, in the solid state they decompose very rapidly, thus, on evaporation of the solutions, they produce alkaline chloride and chlorate. They may be evaluated exactly in the same manner as bleaching powder, since, in this case too, the available chlorine is the valuable constituent.

R. Bauer has proposed the following method for the practical control of the operation of bleaching (*Dingler's Polyt. Journ.*, 1884, 251, 276): A "chlorine tube," 1·5 cm. wide and 50 cms. long and a burette divided into 0·2 c.c. are used. The reagents are hydrochloric acid, solutions of potassium iodide and sodium thiosulphate; 1 c.c. of the latter = 0·001 gramme of chlorine. 10 c.c. of the bleaching liquor are placed in the "chlorine tube," a few c.c. of potassium iodide solution added until, on shaking, no further brown coloration or turbidity occurs; then a few c.c. of hydrochloric acid are added until the turbid solution becomes clear. The thiosulphate solution is now added from the burette, at first rapidly, then in drops, until on gentle shaking the colour becomes paler and finally quite disappears. Each c.c. of thiosulphate solution corresponds to 1 milligramme of chlorine in 10 c.c. of the liquor.

Application.—In bleaching: for many purposes these solutions are said to be more suitable than bleaching powder. They may not be heated. They are more readily washed out and the material may afterwards be given a sulphuric acid bath. They are said also to have a more energetic action than bleaching powder; dilute solutions only should be used. On account of the higher price as compared with bleaching powder they are, however, little used. Alkaline hypochlorites are also added to certain bleaching mixtures, *e.g.*, "chlorozone".

Other Hypochlorites: of aluminium, magnesium, zinc and barium.

The principle of the examination is the same: the active chlorine is estimated. In addition the metal may be determined, since bleaching powder or a cheaper alkaline hypochlorite might be present in large quantity.

Application.—In bleaching special materials. Aluminium hypochlorite, also known as "Wilson's bleaching fluid" for particularly delicate materials; magnesium hypochlorite, "Ramsay's" or "Grouvelle's bleaching fluid" for linen bleaching; zinc hypochlorite is known as "Varrentrapp's bleaching salt". Generally speaking, little used.

Potassium Chlorate, $KClO_3 = 122 \cdot 5$; solubility, cold water $= 7 : 100$; hot water $= 50 : 100$.

The potassium salt is the most important chlorate; it has been largely used since the introduction of aniline black dyeing. The ordinary impurities are metals, earths, chloride, sulphate, nitrate and free chlorine.

A quantitative estimation of the chlorate is generally unnecessary, since the salt is produced in a state of excellent purity. If it is required, the following is the best process: the chlorate is distilled with excess of hydrochloric acid, the chlorine produced led into potassium iodide solution and the liberated iodine titrated with $\frac{1}{10}$ N thiosulphate, 1 c.c. of which = 0·0020416 gramme of $KClO_3$. If, for example, 0·1 gramme of chlorate be taken for the distillation, then, if it is 100 per cent. pure, 48·9 c.c. of $\frac{1}{10}$ N thiosulphate will be required; under these conditions 1 c.c. of $\frac{1}{10}$ N thiosulphate corresponds to 2·045 per cent. of $KClO_3$.

Application.—Almost exclusively as an oxidising agent for aniline black, in producing which it acts as an oxygen carrier in a similar manner to copper sulphide, ammonium vanadate, potassium ferrocyanide, manganates, etc. It has the disadvantage of a low solubility in cold water (7 : 100), on which account it may readily crystallise from strong solutions and cause mishaps in the process (streaks on printed calico). In using concentrated solutions and in printing colours, the employment of sodium chlorate, which is considerably more soluble, prevents these separations. Potassium chlorate is also added to certain bleaching mixtures ("chlorozone"). In the preparation of mordants.

Sodium Chlorate, $NaClO_3$ = 106·5; solubility, cold water = 100 : 100; hot water = 200 : 100.

This salt has the advantages over potassium chlorate of greater solubility and cheapness, however the commercial article is not generally as pure as the potassium salt, and must be carefully examined for impurities. In particular large quantities of chlorides and lime, *i.e.* calcium chloride, are found. The percentage of chlorate may be determined as in the potassium salt.

Application.—Similar to potassium chlorate, when it is required to prepare solutions of greater concentration than 7 : 100; frequently potassium chlorate separates from the printing colour when it is not present even in that theoretical amount.

Aluminium Chlorate, $Al_2(ClO_3)_6 = 555{\cdot}4$.

Is generally prepared by the consumer by double decomposition of potassium chlorate and aluminium sulphate, the solution is then diluted to 20° B. The two component materials (which see) must be examined.

Application.—Has a restricted use for aniline black on woven cotton.

Chromium Chlorate, $Cr(ClO_3)_3 = 302{\cdot}7$.

Is made in a similar manner by the consumer by double decomposition: potassium chromate + potassium chlorate or chrome alum + potassium chlorate. The basic salt is obtained from 975 grammes of neutral chromium chlorate + 75 grammes of freshly precipitated chromium hydroxide. In an examination the proportions of chromium and chloric acid may be estimated.

Application.—Very restricted. In printing, for fixing wood colours (steam black, steam brown).

Aniline Chlorate, see under *Aniline Salts.*

SULPHITE COMPOUNDS.

Sulphurous Acid, Sulphites, Sodium Bisulphite, $SO_2 = 64$. $Na_2SO_3.7H_2O = 252$; solubility, cold water = 25 : 100; hot water = 1 : 1.

These substances are evaluated by the percentage of sulphurous acid, which may be estimated in the aqueous solution of the acid by means of the hydrometer or better—as also in the salts—by volumetric methods.

I. If no other acids are present, titrate with normal caustic soda and a suitable indicator (see Indicators, p. 1 and Table I., also Mohr's *Titrirmethoden*, 6 Aufl., 165; *Dingl. Polyt. Journ.*, 250, 530; Ch. Blarez, *Comptes rend.*, 103, 69). Free sulphurous acid and bisulphites are titrated with normal caustic soda and phenolphthaleïn, the normal sulphites with normal acid and methyl orange. If methyl orange be added to a bisulphite solution, a red coloration indicates an excess of SO_2 over the quantity required to form $NaHSO_3$, which excess may be estimated by titrating with normal soda to the point of neutrality. If, however, the solution is coloured yellow by methyl orange, normal sulphite is present, which may be estimated by titration with normal acid.

II. The most reliable method is Bunsen's iodometric process, when simply the proportion of sulphurous acid is required, without regard to its state of combination or freedom: 5 grammes of the salt (or a corresponding quantity of aqueous sulphurous acid) are dissolved in 1 litre of water, which has been boiled and allowed to cool in a closed vessel. A portion of the solution is measured with a pipette, diluted with boiled water to such an extent—about 1 : 10—that 100 c.c. contain at most 0·05 gramme of SO_2, slightly acidified with sulphuric or hydrochloric acid, a measured excess of $\frac{1}{10}$ N iodine solution added and the excess titrated by $\frac{1}{10}$ N thiosulphate, with starch solution as indicator. 1 c.c. of $\frac{1}{10}$ N iodine solution = 0·0032 gramme of SO_2. Not more than 12 c.c. of iodine solution should be used for 100 c.c. of the sulphite solution titrated.

Application.—In bleaching and as an "antichlor"; for washing material bleached with permanganate in order to remove the manganese peroxide; as a reducing agent; in printing as a solvent for many dye-stuffs (cœruleïn, alizarine blue); many dyes come into the market in the form of double sodium bisulphite compounds. Frequently gaseous sulphur dioxide is used, produced by burning stick sulphur in the sulphur chambers, in which the fabrics to be bleached are hung.

Sodium bisulphite is also known as "leucogen"; more rarely calcium bisulphite is also employed.

The specific gravities of aqueous solutions of sulphur dioxide are given in the following table, together with the strength of the solutions:—

Specific gravity.	SO_2 per cent.	Specific gravity.	SO_2 per cent.	Specific gravity.	SO_2 per cent.	Specific gravity.	SO_2 per cent.
1·0028	0·5	1·0168	3·0	1·0302	5·5	1·0426	8·0
1·0056	1·0	1·0194	3·5	1·0328	6·0	1·0450	8·5
1·0085	1·5	1·0221	4·0	1·0353	6·5	1·0474	9·0
1·0113	2·0	1·0248	4·5	1·0377	7·0	1·0497	9·5
1·0141	2·5	1·0275	5·0	1·0401	7·5	1·0520	10·0

Hydro- or Hypo-sulphurous Acid, Hydro- or Hyposulphites, $H_2O + SO_2 + Zn = ZnSO_3 + H_2$; $H_2 + SO_2 = H_2SO_2$.

Hydrosulphurous acid has recently, in the form of the zinc and calcium salts, attained considerable importance in indigo-dyeing (Schützenberger). It is produced from zinc and bisulphites according to the above equations. The proportion of the bisulphite to the zinc is of certain importance, since the reaction may take place in two ways: $4NaHSO_3 + Zn = ZnSO_3 + Na_2SO_3 + Na_2S_2O_4 + 2H_2O$, and $3NaHSO_3 + Zn = ZnSO_3 + Na_2SO_3 + NaHSO_2 + H_2O$. When lime is added the zinc is precipitated and calcium hydrosulphite results, which has the property of reducing indigo blue, $C_{16}H_{10}N_2O_2$ to indigo white, $C_{16}H_{12}N_2O_2$, containing two hydrogen atoms more. Since these solutions are prepared by the dyer, the quality of the materials and their value must be ascertained.

Application.—In the hydrosulphite vat (more suitable for wool): 30 kilos. of bisulphite solution of 32° to 35° B. + 6 to 7 kilos. of zinc dust are stirred with 30 litres of water; the temperature should not be allowed to exceed 35° C. Then 8 litres of caustic soda solution of 38° B. + 7 kilos. of lime, slaked with 30 litres of water, are added. After standing twelve to twenty-four hours, the clear yellow solution, which can reduce about 10 kilos. of indigo, is drawn off.

Sodium Thiosulphate, "Sodium Hyposulphite," $Na_2S_2O_3 . 5H_2O = 248$; solubility, cold water = 102 : 100; very soluble in hot water.

The impurities may be carbonate, sulphate, sulphite and free alkali. The percentage of thiosulphate is estimated volumetrically by iodine solution: 25 grammes are dissolved to 1,000 c.c., and 25 c.c. of the solution titrated by $\frac{1}{10}$ N iodine solution, with starch solution as indicator. 1 c.c. of iodine solution = 0·024764 gramme of $Na_2S_2O_3 . 5H_2O$.

Application.—In calico-printing to fix metallic oxides; in dyeing as a resist for aniline black; in bleaching to remove the last traces of chlorine from the fibre (hence an "antichlor"); in mordanting silk (6 to 8 per cent. of alum + 4 per cent. of thiosulphate); in dyeing wool with eosine; in washing clothes; for precipitating finely divided sulphur upon wool for methyl and malachite greens (Lauth). Also known under the names of "antichlor" and "hyposulphite of soda," or "hypo".

Miscellaneous Compounds.

Sodium Nitrite, $NaNO_2 = 69$; very soluble in cold and hot water.

This salt has not long been used in the textile industries; its value is estimated by the percentage of nitrite. Heavy metals are frequently found as impurities. The nitrous acid is estimated by oxidation with permanganate, which may be standardised against ferrous ammonium sulphate or silver nitrite, the former compound (also known as Mohr's salt) contains exactly one-seventh of its weight of iron, *i.e.*, 7 grammes of Mohr's salt contain exactly 1 gramme of metallic iron. Good commercial sodium nitrite should contain 95 to 97 per cent. of $NaNO_2$.

About 25 grammes of sodium nitrite are dissolved to 1,000 c.c.; 40 or 45 c.c. of $\frac{1}{5}$ N permanganate solution are added to 100 c.c. of sulphuric acid (1 : 5) and the above nitrite solution somewhat rapidly added at 40° C until decolorisation occurs; then the mixture is titrated back with permanganate until the red coloration remains for five minutes. Lunge's method, according to which the nitrite solution is titrated with permanganate, is unreliable, since in that case some nitrous acid is always lost. $2\ Fe = NaNO_2\ (112 = 69)$.

Calculation: if a grammes of Mohr's salt ($= \frac{a}{7}$ grammes of iron) require b c.c. of permanganate solution, then $112 : 69 = \frac{a}{7} : x$, or $x = \frac{69a}{112 \times 7}$ grammes of $NaNO_2$, which are equivalent to a grammes of Mohr's salt or b c.c. of permanganate solution. Hence 1 c.c. of permanganate solution $= \frac{69a}{112 \times 7 \times b}$ gramme of $NaNO_2$. If now c c.c. of permanganate solution are required in the titration of the nitrite, then in the volume of nitrite solution used there are $\frac{69 \times a \times c}{112 \times 7 \times b}$ grammes of $NaNO_2$, from which the percentage strength of the solid nitrite may be calculated.

If a 1 per cent. nitrite solution (10 grammes in 1,000 c.c.) be used instead of the above, the calculation is simpler: 1 c.c. of permanganate solution = 0·00345 gramme of $NaNO_2$.

Application.—For the so-called diazotised colours, "ice" colours, developed colours, etc. In dyeing and calico printing several of these colouring matters are largely used (*e.g.*, paranitraniline red).

Sodium Phosphate, $Na_2HPO_4 . 12H_2O = 358$; solubility, cold water = 3 : 100 ; hot water = 96 : 100.

Also known as secondary or neutral sodium phosphate. It is often contaminated by sodium chloride, sulphate and carbonate. Its value depends on the percentage of phosphoric acid and the basicity.

Phosphoric acid.—25 grammes are dissolved to 1,000 c.c. ; in 10 or 20 c.c. the phosphoric acid is precipitated with magnesia mixture and ammonia, the precipitate (magnesium ammonium phosphate) filtered after twelve hours, washed with ammoniacal water, dried, ignited and weighed as magnesium pyrophosphate, $Mg_2P_2O_7$.

For the volumetric determination of phosphoric acid by means of uranium solution, see Fresenius, *Quant. Anal.*, ii., 545 ; *Zeits. anal. Chem.*, 1897, xxxvi., 81 ; *J. S. C. I.*, 1897, 262.

Basicity.—If the salt gives an alkaline reaction with phenolphthaleïn it contains more alkali than is required by the formula Na_2HPO_4 ; the excess of alkali may be titrated by normal acid, using phenolphthaleïn as indicator. If no coloration is given by phenolphthaleïn, there is present the neutral salt alone or a mixture of Na_2HPO_4 and NaH_2PO_4, the latter of which may be estimated by titration with caustic soda and phenolphthaleïn, the former by titration with acid and methyl orange.

Application.—In Turkey red dyeing instead of cow and sheep dung (which are now almost entirely replaced by sodium phosphate, arsenate and silicate) ; with wood colours (a more brilliant black) ; in silk-dyeing for weighting and to fix the tin and iron (4° to 6° B.) instead of, or together with soda ; sodium phosphate is better than soda for weighting purposes, but it does not make the fibre voluminous, and overloading the fibre with phosphoric acid may be harmful. In dyeing with azo dye-stuffs. In recent years ammonium phosphate has been successfully employed in place of the sodium salt.

Water Glass, Potassium or Sodium Silicate, $K_2Si_4O_9$ = 334, $Na_2Si_4O_9$ = 302.

Since water glass frequently comes into commerce in solution, its value may be directly determined by means of the hydrometer; the silica may also be estimated (Fresenius, *Quant. Anal.*, i., 347). About 50 grammes are dissolved to 1,000 c.c., 50 c.c. of the solution repeatedly evaporated with hydrochloric acid and then heated for some hours at 110° C. The silicic acid then becomes insoluble, it is moistened with hydrochloric acid, filtered off, dried, ignited and weighed as SiO_2.

Silicic acid may also be determined acidimetrically by conversion into potassium fluosilicate (Stolba, *Zeits. anal. Chem.*, iv., 163), or directly titrated, with methyl orange as indicator, by normal acid.

Commercial water glass frequently contains a considerable excess of caustic soda, which would be very harmful to animal fibres and must, as far as possible, be absent; caustic soda would also have a solvent action on alumina mordants.

Application.—Similar to sodium phosphate; also in bleaching with hydrogen peroxide (instead of ammonia); in silk-dyeing in place of borax (Alkali Blue, logwood); as a mordant for cotton (water glass + H_2SO_4 gives SiO_2, which fixes basic dyes); for fire-proofing fabrics; as an addition to soap; as an addition to varnishes; for dressing cotton warp; in printing, as a substitute for albumin; in finishing; as a fixing agent for alumina mordants and tin (silk); to produce lustrous and tensile effects; since 1893 used in silk-dyeing to produce the important zinc phosphate-silicate weighting.

Sodium Arsenate, $Na_2HAsO_4 . 12H_2O$ = 402; solubility, cold water = 28 : 100, very soluble in hot water.

It must be free from nitric acid, not contain much lime, and at most contain 1 per cent. of As_2O_3.

The *basicity* may be estimated by direct titration with normal acid and methyl orange, supposing that no other acids are present. 1 c.c. of normal acid = 0·312 gramme of $Na_2 HAsO_4 . 7H_2O$.

Arsenic Acid.—0·5 gramme of the sample is precipitated as magnesium ammonium arsenate (Fresenius, *Quant. Anal.*, i., 283), the precipitate brought on to a weighed filter, dried at 100° C. and weighed. The precipitate may also be ignited and estimated as magnesium pyro-arsenate.

Having regard to the restricted use of this salt, the various methods of estimation are not described; the principle of the method given by Sutton (*Volumetric Analysis*, sixth ed.) may be mentioned—the arsenic acid is reduced to arsenious acid and the latter titrated with iodine solution.

Arsenious acid is determined by a reversal of the process for chlorine and iodine by means of sodium arsenite (see *Bleaching Powder*). 5 to 10 grammes of the arsenate are dissolved, bicarbonate solution added and the liquid titrated with $\frac{1}{10}$ N iodine solution, 1 c.c. of which = 0·00495 gramme of As_2O_3. More than 1 per cent. of arsenious acid should not be present (see Christensen, *Zeits. anal. Chem.*, 1897, xxxvi., 81; *J. S. C. I.*, 1897, 262).

Application.—Restricted on account of its poisonous nature, otherwise similar to sodium phosphate.

Sodium Tungstate, $Na_6W_7O_{24} . 16H_2O = 1854·5$, has now little interest. The tungstic acid may be determined by the method given by Fresenius (*Quant. Anal.*, ii., 427). On account of the unimportance of the compound, the method is not described here.

According to H. Silbermann (*Färber-Zeit.*, 1897, No. 5, 70) the tungstic acid is estimated by precipitating the solution of a weighed quantity of sodium tungstate with excess of hydrochloric acid, digesting for some hours at 30° to 40° C., washing the precipitate first with dilute hydrochloric acid, then with water, drying at 105° C. and weighing.

Application.—It possesses historic interest as an oxidising agent (instead of copper sulphide), chromium tungstate was also used; also as a weighting agent for silk, fixed upon the fibre as iron or tin tungstate.

Sodium Stannate, Preparing Salt, $Na_2\,SnO_3.3H_2O = 266$ 44·4 per cent. of tin.

Commercial sodium stannate contains 30 to 44 per cent. of stannic oxide; its impurities are caustic soda, soda, common salt and iron, occasionally it is adulterated with arsenate and tungstate. It should dissolve with as little residue as possible, be free from iron and not be too alkaline.

The total alkali is determined by titration with normal acid and methyl orange.

T. Goldschmidt estimates the tin as follows: 50 grammes of the sample are dissolved to 500 c.c., 10 c.c. of the solution are reduced in the presence of hydrochloric acid. The separated tin is dissolved in the same flask in an atmosphere of carbon dioxide, by boiling and adding more hydrochloric acid. After cooling, the stannous chloride formed is determined by means of standard ferric chloride solution and potassium iodide (starch solution). The titration is also conducted whilst a current of carbonic acid is led into the flask.

Application.—As a mordant for azo dyestuffs (4° to 5° B.), followed by a bath of neutralised alum (25 grammes of alum + 10 grammes of crystallised soda in 1,000 c.c.); it was formerly used for weighting silk, but has now been entirely replaced by stannic chloride; in printing on cotton and wool.

Sodium Aluminate, $Na_2Al_2O_4 = 164$.

When this compound is titrated with acid and phenolphthaleïn (less well with litmus), the end-reaction appears when all the alkali is saturated and the alumina has separated; if methyl orange be used, the end-reaction occurs when the compound $Al_2(SO_4)_3$ has been formed, and the separated alumina has redissolved. 20 grammes of sodium aluminate are dissolved to 1,000 c.c.; 10 c.c. are titrated quite hot (in order to avoid the influence of carbonic acid, if present) with $\frac{1}{5}$ N hydrochloric acid and phenolphthaleïn until the colour disappears; methyl orange is then added and titration with the same acid continued until the red coloration appears. The difference gives the alumina. (See also Lunge, *Zeits. angew. Chem.*, 1890, 227, 293; *J. S. C. I.*, 1890, 767.) 1 c.c. of $\frac{1}{5}$ N hydrochloric acid = 0·0062 gramme of Na_2O = 0·0034 gramme of Al_2O_3.

Example: 0·2 gramme of aluminate require 8·5 c.c. (phenolphthaleïn) and 15·35 c.c. (methyl orange) of $\frac{1}{5}$ N hydrochloric acid, a total of 23·85 c.c. The

$$Na_2O = \frac{8{\cdot}5 \times 0{\cdot}0062 \times 100}{0{\cdot}2} = 26{\cdot}35 \text{ per cent., the}$$

$$Al_2O_3 = \frac{15{\cdot}35 \times 0{\cdot}0034 \times 100}{0{\cdot}2} = 26{\cdot}095 \text{ per cent.}$$

Application.—Very restricted in calico-printing.

Borax, Sodium Biborate, $Na_2B_4O_7.10H_2O = 382$; solubility, cold water = 6 : 100; hot water = 200 : 100.

The boric acid is determined by titration with acid and methyl orange. 25 grammes are dissolved to 1,000 c.c. and 50 c.c. titrated with normal sulphuric acid and methyl orange until the red coloration appears. 1 c.c. of normal acid = 0·191 gramme of crystallised borax. Soda, sodium chloride and sulphate occurs as impurities. The percentage of soda, which would be titrated along with the borax, must be deducted when it is appreciable (*Zeits. angew. Chem.*, 1896, xxii., 679; *Zeits. anal. Chem.*, 1897, 568).

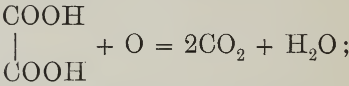
Application.—Instead of soda in fermenting logwood; in calico-printing, as a solvent for caseïn; in dyeing wool with

Alkali Blue (10 per cent. of the weight of the wool, or 300 to 350 grammes per 100 litres of bath).

Potassium Permanganate, $KMnO_4 = 158$; solubility, cold water – 6·45 : 100 ; very soluble in hot water.

The percentage of pure permanganate is determined volumetrically against oxalic acid or a ferrous salt. A solution of 6·32 grammes of pure permanganate in 1,000 c.c. would give a $\frac{1}{5}$ N solution. Accordingly 20 c.c. of normal oxalic acid, or 100 c.c. of $\frac{1}{5}$ N oxalic acid, would require exactly 100 c.c. of the permanganate solution, if it were made from the chemically pure salt. The calculation is very simple : if 105 c.c. of permanganate are required instead of 100 c.c. for 100 c.c. of $\frac{1}{5}$ N oxalic acid, then the permanganate is 94·85 per cent. pure (105 : 100 = 100 : x, x = 94·85).

The oxalic acid is strongly acidified with sulphuric acid and the titration conducted at 60° to 70° C. until a permanent red coloration appears. $2KMnO_4 + 3H_2SO_4 = K_2SO_4 + 2MnSO_4 + 3H_2O + 5O$;



$$\begin{array}{c}COOH\\ |\\ COOH\end{array} + O = 2CO_2 + H_2O$$

; (thus 2 mols. of permanganate correspond to 5 mols. of oxalic acid). The solution is standardised in a similar manner against a ferrous salt, *e.g.*, ferrous ammonium sulphate (Mohr's salt), 7 grammes of which = 1 gramme of iron ; $5Fe = 1KMnO_4$; $2FeO + O = Fe_2O_3$. 1 c.c. of $\frac{1}{5}$ N permanganate solution = 0·0112 gramme of metallic iron = 0·0144 gramme of FeO = 0·016 gramme of Fe_2O_3.

Application.—In bleaching wool (neutral, alkaline or acid bath ; best in a neutral bath of 1 : 1,000, since the bath may always be used again ; in an alkaline bath the bleaching is slower, in an acid bath quicker). After bleaching, the material is washed with sulphurous acid, generally with an addition of sulphuric acid (occasionally 2 to 3 mols., otherwise the material may readily turn brown). Potassium permanganate cannot be used for bleaching silk, since the fibre is injured.

Potassium Bichromate, Sodium Bichromate, $K_2Cr_2O_7 = 295$; 68 per cent. of CrO_3; solubility, cold water $= 10{\cdot}4 : 100$; hot water $= 1 : 1$.—$Na_2Cr_2O_7 . 2H_2O = 299$: 67·19 per cent. of CrO_3; more soluble than the potassium salt.

Potassium bichromate often contains potassium sulphate, sodium sulphate and normal chromate. Most important is the percentage of chromic acid.

Total chromic acid is determined by volumetric methods against ferrous ammonium sulphate. The solution of potassium or sodium bichromate, acidified with sulphuric acid, is mixed with an excess of solution of ferrous ammonium sulphate, and the excess determined by adding $\frac{1}{10}$ N bichromate solution until a drop of the solution no longer gives a blue coloration (Turnbull's blue) with potassium ferricyanide on a porcelain plate. $CrO_3 = 3FeO$. Knecht, Rawson and Löwenthal give the following process: 5 grammes of the sample are dissolved to 1,000 c.c., and a burette filled with the solution. (In the case of the sodium salt it is better to dissolve 25 grammes to 500 c.c., and again dilute 100 c.c. to 1 litre.) 1 gramme of pure crystallised ferrous ammonium sulphate is weighed off, dissolved in a porcelain dish in a little water and 50 c.c. of 10 per cent. sulphuric acid added. The bichromate solution is added from the burette, with constant stirring, until a drop of the solution mixed with potassium ferricyanide solution on a porcelain plate no longer gives a blue or greenish-blue coloration. 1 gramme of ferrous ammonium sulphate reduces 0·0853 gramme of CrO_3, therefore the volume of chromate solution added from the burette contains 0·0853 gramme of CrO_3.

Example: 5 grammes of $K_2Cr_2O_7$ in 1,000 c.c.; 1 gramme of ferrous ammonium sulphate requires 25·2 c.c. of this solution; 25·2 : 0·0853 = 1,000 : x; x = 3·385 grammes of CrO_3; 5 : 3·385 = 100 : x; x = 67·70 per cent. of CrO_3.

Free chromic acid is detected by means of the hydrogen peroxide reaction: 2·5 to 5 grammes of the bichromate are dissolved in 40 to 50 c.c. of water, 10 c.c. of hydrogen peroxide and 20 c.c. of ether added, and the whole mixed in a cylinder. In the presence of free chromic acid the ether layer becomes blue; the coloration rapidly disappears.

Neutral chromate is estimated by McCulloch by means of the above reaction (*Chem. News*, 1887, lv., 2). As in the detection of free chromic acid, 2·5 to 5 grammes are dissolved in 40 to 50 c.c. of water and the solution shaken in a stoppered cylinder of 120 c.c. capacity with 10 c.c. of hydrogen peroxide and 20 c.c. of ether. Decinormal sulphuric acid is now added in small quantities at a time, with frequent shaking, until the ether acquires a pale blue colour. 1 c.c. of $\frac{1}{10}$ N acid = 0·01003 gramme of CrO_3 as normal chromate, 0·01623 gramme of Na_2CrO_4 or 0·01943 gramme of K_2CrO_4, in the quantity of material taken. The hydrogen peroxide and bichromate naturally should not contain free acid, any free acid they contain must be deducted.

Bichromate.—(*a*) According to McCulloch the bichromate may be directly estimated by titration with normal caustic soda and phenolphthaleïn; the normal chromate may then be found by deducting the chromic acid present as bichromate from the total chromic acid. Since the end-reaction in titrating with caustic soda is ill defined, excess is added and titrated back with normal acid. 1 c.c. of $\frac{1}{10}$ N caustic soda = 0·01003 gramme of CrO_3 as bichromate.

(*b*) R. Thomson (*Chem. News*, 1885, lii., 29) titrates directly with lacmoid as indicator (see under *Indicators*), towards which bichromate is neutral, but normal chromate alkaline. The latter can accordingly be estimated by means of standard acid and lacmoid paper.

Example: 100 c.c. of 1 per cent. solution (= 1 gramme), in titration with $\frac{1}{10}$ N sulphuric acid and lacmoid required 12·6 c.c. of acid; 0·01003 × 12·6 × 100 = 12·64 per cent of CrO_3 as chromate. Total CrO_3 = 70·60 per cent., therefore bichromate = 70·60 – 12·64 = 57·96 per cent. of CrO_3 as bichromate.

Application.—One of the most important salts used in dyeing. As a mordant for wool (with tartar, oxalic acid, sulphuric acid, lactic acid, etc.); in developing aniline black (dyeing and printing, also lead chromate in printing); in blue-printing; for fast browns from catechu; as an addition to bluestone black (logwood), which is thus made more resistant to alkalis; as a discharging agent in calico-printing; for chrome yellow, orange and olive; in combination with the indigo vat as "Jäger" green.

Sodium bichromate is hygroscopic, generally more impure—contains more normal chromate—and requires more careful control, whilst the potassium salt is generally obtained in very good quality. Also the sodium salt is used with reluctance in printing, since the goods dry badly, in wool-dyeing, on the other hand, it is preferred because it is cheaper and more easily soluble.

Vanadates.—Ammonium vanadate $(NH_4)_3VO_4$ = 169. Vanadium chloride, blue vanadium solution (= vanadium tetra-, tri- and bichlorides).

The vanadates are evaluated by means of the proportion of vanadic acid, which can be determined (Fresenius, *Quant. Anal.*, ii., 312) by precipitating ammonium vanadate by saturated ammonium chloride solution, when impurities remain in solution. The precipitate is filtered, ignited, and weighed as vanadic acid, V_2O_5. The most usual impurity is chromic acid.

Application.—As an oxidising agent in printing aniline black, in place of copper sulphide, which readily dries and causes streaks. It is active at extraordinary dilutions—0·2 gramme per kilo. of printing colour; excess may readily spoil the fabric. The blue vanadium solution is obtained by reducing vanadic acid with glycerin in the presence of hydrochloric acid at water-bath temperatures (100 grammes of ammonium vanadate + 400 grammes of hydrochloric acid + 400 grammes of water, then a solution of about 50 grammes of glycerin in 100 c.c. of water added, the mixture warmed until the blue coloration appears and finally diluted to 10 litres).

Alkalis.

Ammonia, $NH_3 = 17$; solubility, cold water = 1,050 volumes : 1. The strength is estimated by means of the hydrometer, or better volumetrically. Fifty grammes are diluted to 1,000 c.c. and 50 c.c. titrated with normal acid and litmus, etc. 1 c.c. = 0·017 gramme of NH_3.

Ammonia solution should leave no, or a very slight, residue on evaporation. The common impurities are sulphuric acid, sulphuretted hydrogen, carbonic acid and chlorine. Ordinary technical ammonia = 16° B. = 9·9 per cent of ammonia.

Aqueous Ammonia Solution (Lunge).

Specific gravity.	NH_3 per cent.	Specific gravity.	NH_3 per cent.	Specific gravity.	NH_3 per cent.	Specific gravity.	NH_3 per cent.
0·996	0·91	0·968	7·82	0·952	12·17	0·924	20·49
0·992	1·84	0·964	8·84	0·950	12·74	0·920	21·75
0·988	2·80	0·962	9·35	0·946	13·88	0·916	23·03
0·984	3·80	0·960	9·91	0·942	15·04	0·910	24·99
0·980	4·80	0·958	10·47	0·938	16·22	0·900	28·33
0·976	5·80	0·956	11·03	0·934	17·42	0·890	31·75
0·972	6·80	0·954	11·60	0·930	18·64	0·882	34·95

Application.—For washing wool and shoddy dyed with alizarine (the dye becomes faster to rubbing); in bleaching with hydrogen peroxide; for precipitating lead on the fibre; instead of soda and fixed alkalis.

Ammonium Salts.—Sulphate, acetate, oxalate.

The ammonia is estimated in the ordinary manner by distillation: 0·5 to 1 gramme of the sample is distilled with excess of caustic soda, the receiver contains a measured excess of normal acid, which is titrated, after the distillation, by normal caustic soda. 1 c.c. of N acid used = 0·017 gramme of NH_3 (Fresenius, *Quant. Anal.*, i., 178).

Application.—Restricted on account of the high price. In dyeing with sulphonic acid dye-stuffs to secure better equalisation (induline).

Sodium Hydroxide, Caustic Soda, $NaOH = 40$; solubility, cold water = 60 : 100; hot water = 210 : 100.

Caustic soda comes into the market both solid and in solution, the latter at strengths of 30°, 36° and 39° B. The value is given by the percentage of sodium hydroxide, which can be determined in solutions approximately by means of the hydrometer, more acurately by titration. Caustic soda should, as far as possible, be free from carbonate, thus an estimation of sodium carbonate is important. Either the caustic soda alone is determined, or the carbonate also.

Total Alkali.—25 grammes are dissolved to 1,000 c.c. and 100 c.c. titrated with N acid and methyl orange, phenolphthaleïn, etc. Also, although more complicated, excess of acid may be added, the liquid boiled for 5 minutes and titrated back with N caustic soda, litmus or phenolphthaleïn being the indicator. 1 c.c. of normal acid = 0·04 gramme of NaOH or 0·031 gramme of Na_2O.

Caustic Alkali.—To a second 100 c.c. excess of neutral barium chloride solution is added to precipitate the carbonate, and the mixture titrated (without filtering) with

Specific Gravity of Caustic Soda Solutions at 15° (Lunge)

Specific gravity.	Degrees Baumé.	Degrees Twaddell.	Na_2O per cent.	Na OH per cent.	1 cub. metre contains	
					Na_2O Kilos.	Na OH Kilos.
1·007	1	1·4	0·47	0·61	4	6
1·014	2	2·8	0·93	1·20	9	12
1·022	3	4·4	1·55	2·00	16	21
1·029	4	5·8	2·10	2·71	22	28
1·036	5	7·2	2·60	3·35	27	35
1·045	6	9·0	3·10	4·00	32	42
1·052	7	10·4	3·60	4·64	38	49
1·060	8	12·0	4·10	5·29	43	56
1·067	9	13·4	4·55	5·87	49	63
1·075	10	15·0	5·08	6·55	55	70
1·083	11	16·6	5·67	7·31	61	79
1·091	12	18·2	6·20	8·00	68	87
1·100	13	20·0	6·73	8·68	74	95
1·108	14	21·6	7·30	9·42	81	104
1·116	15	23·2	7·80	10·06	87	112
1·125	16	25·0	8·50	10·97	96	123
1·134	17	26·8	9·18	11·84	104	134
1·142	18	28·4	9·80	12·64	112	144
1·152	19	30·4	10·50	13·55	121	156
1·162	20	32·4	11·14	14·37	129	167
1·171	21	34·2	11·73	15·13	137	177
1·180	22	36·0	12·33	15·91	146	188
1·190	23	38·0	13·00	16·77	155	200
1·200	24	40·0	13·70	17·67	164	212
1·210	25	42·0	14·40	18·58	174	225
1·220	26	44·0	15·18	19·58	185	239
1·231	27	46·2	15·96	20·59	196	253
1·241	28	48·2	16·76	21·42	208	266
1·252	29	50·4	17·55	22·64	220	283
1·263	30	52·6	18·35	23·67	232	299
1·274	31	54·8	19·23	24·81	245	316
1·285	32	57·0	20·00	25·80	257	332
1·297	33	59·4	20·80	26·83	270	348
1·308	34	61·6	21·55	27·80	282	364
1·320	35	64·0	22·35	28·83	295	381
1·332	36	66·4	23·20	29·93	309	399
1·345	37	69·0	24·20	31·22	326	420
1·357	38	71·4	25·17	32·47	342	441
1·370	39	74·0	26·12	33·69	359	462
1·383	40	76·6	27·10	34·96	375	483
1·397	41	79·4	28·10	36·25	392	506
1·410	42	82·0	29·05	37·47	410	528
1·424	43	84·8	30·08	38·80	428	553
1·438	44	87·6	31·00	39·99	446	575
1·453	45	90·6	32·10	41·41	466	602
1·468	46	93·6	33·20	42·83	487	629
1·483	47	96·6	34·40	44·38	510	658
1·498	48	99·6	35·70	46·15	535	691
1·514	49	102·8	36·90	47·60	559	721
1·530	50	106·0	38·00	49·02	581	750

normal acid and methyl orange or litmus to incipient red coloration. 1 c.c. of N acid = 0·04 gramme of NaOH or 0·031 gramme of Na_2O *as free caustic soda.*

Sodium Carbonate.—The difference between the total alkali and caustic alkali gives the carbonate.

Commercial caustic soda generally contains much common salt, sodium sulphate and excess of water, to the extent of 30 per cent. and more.

Application.—Restricted. In the preparation of Turkey red oil; for washing and bleaching purposes; in printing indigo; as an addition to the potassium ferricyanide oxidising mass. Caustic potash, since it is dearer and without special advantage, is hardly used in the textile industries. See also *Zeits. angew., Chem.*, 1896, 455.

Lime.—Quicklime, CaO = 56; solubility, cold water = 1 : 778; hot water = 1 : 1,270. Slaked lime $Ca(OH)_2$ = 74.

The amount of calcium oxide is best estimated by the process given by Degener: 100 grammes of quicklime are completely slaked, the paste brought into a 500 c.c. flask, the liquid filled up to the mark, and after shaking, 100 c.c. withdrawn with a pipette. This 100 c.c. is again diluted to 1,000 c.c., well shaken, and 25 c.c. brought into a porcelain dish. A few grains of precipitated calcium carbonate are then added and alcoholic phenacetolin solution; normal acid is now added, under constant stirring, until the liquid where the acid drops in, which becomes yellow, at once turns red again. When the red coloration does not appear until after some seconds, the volume of acid used is read off, and two more drops added. If the liquid now remains yellow the previous reading was correct, but if it again becomes red, the reading was premature and the addition of acid must be continued. 1 c.c. of N acid = 0·028 gramme of CaO.

The powder formed on slaking should be fine and pulverulent, feel soft, and when stirred with a small quantity of water should give a smooth, "fat," and slippery paste. If this is not the case the lime is bad, "lean," and contains much magnesia and alumina.

Application.—In vat-dyeing to dissolve the indigo; in black cotton-dyeing (the cheapest black); practically no longer used for bleaching; to strip wood colour from wool, etc.

Sodium Carbonate, Soda, Calcined soda $Na_2CO_3 = 106$; solubility, cold water = 7 : 100; hot water 45 : 100. Crystallised soda or soda crystals, $Na_2CO_3 . 10H_2O = 286$; solubility, cold water = 21·3 : 100; hot water = 420 : 100.

Soda is usually contaminated by sulphate, chloride, sulphite, alkaline sulphide (see sodium nitroprusside), lime, iron and free caustic soda. Most important are estimations of the total alkali and caustic soda.

Total Alkali.—The estimation is conducted as in caustic soda: 25 to 50 grammes are dissolved to 1,000 c.c. (25 grammes of calcined, 50 grammes of crystallised soda) and 50 c.c. of the solution titrated with N acid, methyl orange being the indicator. 1 c.c. of N acid = 0·031 gramme of Na_2O = 0·053 gramme of Na_2CO_3 = 0·143 gramme of $Na_2CO_3 . 10H_2O$. The value obtained corresponds to the total alkali, from which the caustic alkali, if determinable, to be deducted.

Caustic Alkali.—Excess of barium chloride solution is added to a second 50 c.c. and the solution tested, after a few minutes, for alkaline reaction. If it is alkaline, it is titrated with $\frac{1}{10}$ N hydrochloric or sulphuric acid, and methyl orange (or litmus) to acid reaction. 1 c.c. of $\frac{1}{10}$ N acid = 0·0031 gramme of Na_2O.

Sodium Carbonate.—The difference between the total and caustic alkali gives the soda present as carbonate.

Bicarbonate, which may be present, is not generally estimated, since it does not produce any inconveniences. The iron is often harmful; it is best estimated by colorimetric methods. (Estimation of bicarbonate, see Lunge, *Handbuch der Sodaindustrie*, ii., 648; Böckmann, *Chemisch-technische Untersuchungen*, i., 390, 391.)

Application.—In wool-washing to remove grease (caustic alkali harmful); in calico-bleaching; in linen-bleaching; for fixing metallic oxides on the fibre (*e.g.*, tin oxides on silk); in alizarine-dyeing for softening and fixing alumina and chromium oxide on the fibre; to neutralise alum (Turkey red); in dyeing cotton with bluestone (logwood) black; in dyeing with the benzidine dyes, etc.

For the same reason that caustic potash is not used, the dearer potassium carbonate (potashes) has no advantages over soda, which would justify its employment.

Calcium Carbonate, Chalk, $CaCO_3 = 100$; solubility, cold water = 1 : 10,600; hot water = 1 : 8834.

The pure compound is found in commerce. The proportion of calcium carbonate may be estimated as in soda; an excess of acid is naturally added, which is titrated back. Since a solution cannot be made, 2 grammes are weighed off, 50 c.c. of N acid cautiously added (the substance should previously be covered with water), the carbonate carefully dissolved and the solution titrated, with or without previous boiling to drive off carbonic acid, by means of N caustic soda and phenolphthaleïn or methyl orange respectively. 1 c.c. of N acid = 0·05 gramme of $CaCO_3$.

Application restricted. For fixing metallic oxides (Turkey red dyeing); for sumach black; for removing traces of acid from fabrics, etc.

PEROXIDES.

Hydrogen Peroxide, $H_2O_2 = 34$; very readily soluble in water. The value of the solution depends on the percentage of hydrogen peroxide and the absence of mineral acids—hydrochloric and sulphuric. It generally comes into commerce as a 3 per cent. solution, the strength of which is estimated by one of the following processes:—

I. With permanganate. 10 c.c. of the solution are diluted, 30 c.c. of sulphuric acid (1 : 3) added and the liquid titrated in the cold by potassium permanganate, until the red coloration is just permanent. 1 c.c. of $\frac{1}{5}$ N permanganate $= 0{\cdot}0034$ gramme of $H_2O_2 = 0{\cdot}0016$ gramme of O.

II. By means of potassium iodide and thiosulphate (see Kingzett, *Journ. Chem. Soc.*, 1880, 792). 10 c.c. are mixed with 30 c.c. of dilute sulphuric acid and excess of potassium iodide. After five minutes the liberated iodine is determined by titration with $\frac{1}{10}$ N thiosulphate solution and starch, until the blue coloration disappears. 1 c.c. of $\frac{1}{10}$ N thiosulphate $= 0{\cdot}0017$ gramme of $H_2O_2 = 0{\cdot}0008$ gramme of O. (1 gramme of oxygen at 0° C. and 760 mm. pressure occupies 697·5 c.c.).

III. Coulamine recommends a more complicated gasometric estimation, unsuitable for technical purposes (*Dingler's polyt. Journ.*, 1888, 267, 238).

The solution should also be examined for residue, hydrochloric acid and especially sulphuric acid.

Application.—For bleaching tussur, chappe,[1] pure and mixed silk fabrics, wool and jute; also in combination with sulphurous acid (first H_2O_2, then SO_2, or the reverse); as an oxidising agent it has not yet found application in the textile industries. As a bleaching agent it is being supplanted by sodium peroxide.

Barium Peroxide, $BaO_2 = 169$; insoluble in cold, decomposed by hot water.

The peroxide always contains much monoxide; the value is determined by the proportion of peroxide. This is best estimated as follows (*Zeits. anal. Chem.*, 1892, xxxi., 28):

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[1] A term used on the Continent for silk waste which has not been boiled to remove the gum.

0·3 gramme of barium peroxide is suspended in 500 c.c. of water and then dissolved by the addition of 10 c.c. of hydrochloric acid; 10 c.c. of 20 per cent. manganous sulphate solution are next added and the liquid titrated by $\frac{1}{5}$ N permanganate, 1 c.c. of which = 0·0169 gramme of BaO_2. In order to obtain a good average result, it is necessary to perform at least three to four titrations and take the mean. Good technical barium peroxide should contain at least 80 per cent. of BaO_2.

Application.—As a bleaching agent, followed by a bath of hydrochloric acid (most suitable for tussur, chappe and various woollen materials). Wool generally loses in elasticity by treatment with hydrogen peroxide (5 to 10 per cent. of barium peroxide suspended in water, the materials left in for one to two hours and then given a dilute hydrochloric acid bath). The action is rather more energetic than that of ready-made hydrogen peroxide, since the latter substance comes into action *in statu nascendi.*

Sodium Peroxide, $Na_2O_2 = 78$; decomposed by water. The proportion of sodium peroxide is determined in a similar manner to barium peroxide. The weighing and other operations must be conducted more quickly and carefully, since sodium peroxide is rapidly decomposed in moist air. The peroxide must also be dissolved in a large volume of dilute sulphuric acid (about equal to that of the hydrochloric acid mentioned above), otherwise loss occurs; the solution is then titrated as above with $\frac{1}{5}$ N permanganate, 1 c.c. of which = 0·0078 gramme of Na_2O_2.

Application.—Has already found extensive application as a bleaching agent. Especially suitable for chappe, tussur and straw.

Zinc Dust, $Zn = 65$; insoluble in water.

Zinc dust should contain no grains which can be seen or felt by the fingers, it should feel uniformly dusty. Also it should not contain too much iron and zinc oxide, the usual impurities. The proportion of metallic zinc is estimated by means of its reducing properties (see also Drewson, *Zeits. Anal. Chem.*, xix., 50, and Fresenius, *Quant. Anal.*, ii., 288). 10 grammes of zinc dust are brought into a conical flask with 40 grammes of potassium bichromate and 300 c.c. of water, 150 c.c. of sulphuric acid (1 : 5) are gradually added during three to four hours with frequent stirring on the water-bath. The addition of acid must be so regulated that no evolution of hydrogen is perceptible throughout, *i.e.* the whole of the hydrogen produced must reduce the bichromate whilst *in statu nascendi ;* 5 to 10 c.c. (as a maximum) of acid are added every ten minutes. When the zinc is completely dissolved the solution is diluted to 1,000 c.c. and 100 c.c. titrated with a solution of ferrous ammonium sulphate (70 : 500) standardised against bichromate solution (15 : 1000), until a drop of the mixture begins to give a blue coloration with potassium ferricyanide on a porcelain plate. About 150 c.c. of ferrous solution will be required, this corresponds to about 90 per cent. of metallic zinc, which is the percentage in good zinc dust.

Calculation : a grammes of $K_2Cr_2O_7$ in 1,000 c.c., b c.c. of ferrous ammonium sulphate solution $= d$ c.c. of $K_2Cr_2O_7$ solution. To 100 c.c. of the above solution to be titrated (*i.e.*, when w grammes of substance were taken, to $\frac{w}{10}$ grammes of substance), e c.c. of ferrous ammonium sulphate solution were added, and f c.c. of bichromate solution were then required. The e c.c. of ferrous solution $= \frac{de}{b}$ c.c. of $K_2Cr_2O_7$ solution, accordingly in the 100 c.c. of solution there is an excess of $(\frac{de}{b} - f)$ c.c. of $K_2Cr_2O_7$ solution $= (\frac{de}{b} - f)\,\frac{a}{1000}$ grammes of $K_2Cr_2O_7$. Thus $\frac{w}{10}$ grammes of zinc dust have reduced (if k grammes of $K_2Cr_2O_7$ were dissolved) $\frac{k}{10} - (\frac{de}{b} - f)\,\frac{a}{1000}$ grammes of $K_2Cr_2O_7$. The weight of the potassium bichromate, multiplied by 0·6619, gives the equivalent weight of zinc, or 1 c.c. of N bichromate solution = 0·0325 gramme of Zn. ($3Zn + 7H_2SO_4 + K_2Cr_2O_7 = Cr_2(SO_4)_3 + 3ZnSO_4 + K_2SO_4 + 7H_2O$; $3Zn = 1K_2Cr_2O_7$ or 195 Zn = 294·95 $K_2Cr_2O_7$).[1]

Knecht and Rawson work in the following manner: 0·662 gramme of zinc dust (the same quantity of pure zinc would reduce exactly 1 gramme of $K_2Cr_2O_7$) is mixed with 80 c.c. of a bichromate solution containing 25 grammes in 1 litre, and with 10 c.c. of dilute sulphuric acid; after ten to fifteen minutes, a second similar quantity of acid is added and later a third, stirring from time to time. Then 20 c.c. of strong sulphuric acid are added and an excess (about 10 grammes) of pure ferrous ammonium sulphate. After mixing, a drop of the solution must give a blue coloration with potassium ferricyanide, otherwise more ferrous salt must be added. The excess of ferrous salt is then titrated by N bichromate solution. The quantity of bichromate, reduced by 0·662 gramme of the sample, when multiplied by 100 gives immediately the percentage of metallic zinc. Any iron present will be included in the zinc. The weight of pure zinc is found by multiplying by 0·6619 the weight of the bichromate reduced by it; in calculating in this manner it is not necessary to weigh off exactly a definite quantity of zinc dust.

Example: 0·662 gramme of zinc dust; 80 c.c. of bichromate solution (25 : 1000); 10 grammes of ferrous ammonium sulphate; 6·1 c.c. of bichromate solution in titrating back

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[1] The calculation is evidently based upon a method of operation slightly different to that described in the previous paragraph, namely, that to the 100 c.c. of solution, after reduction of the bichromate by the zinc dust, an excess (*e* c.c.) of ferrous ammonium sulphate solution is added and the excess titrated by adding *f* c.c. of bichromate solution until the blue coloration with potassium ferricyanide is no longer given (Translator).

(1 gramme of the ferrous salt = 0·1253 gramme of $K_2Cr_2O_7$); 0·025 × 86·1 = 2·1525 = total $K_2Cr_2O_7$; 0·1253 × 10 = 1·253 grammes of $K_2Cr_2O_7$ reduced by the ferrous salt; 2·1525 – 1·253 = 0·8995 gramme of $K_2Cr_2O_7$ reduced by the zinc; 0·8995 × 100 = 89·95 per cent. of metallic zinc. Or 0·662 gramme of zinc dust = 0·8995 gramme of $K_2Cr_2O_7$, 0·8995 × 0·6619 = 0·5957 gramme of metallic zinc in 0·662 gramme of the sample, hence 89·95 per cent. of metallic zinc.

Liebschütz first withdraws the iron from 1 gramme of the sample by means of a magnet, and then treats with a warm neutral solution of 15 grammes of copper sulphate. After standing some time the oxide is removed by dilute sulphuric acid, the separated metallic copper washed with water, dissolved in nitric acid and titrated by normal potassium cyanide solution. The weight of the copper so found, multiplied by 1·027, gives the weight of metallic zinc in the quantity taken.

The metallic zinc may also be estimated gasometrically by means of the volume of hydrogen evolved on treating the zinc dust with dilute hydrochloric or sulphuric acid.

Application.—As a reducing agent and resist in calico-printing; in indigo-dyeing (hydrosulphite vat).

CHAPTER IV.

ORGANIC COMPOUNDS.

Fatty Acids and Their Salts.

Acetic Acid, $CH_3.CO.OH = 60$; miscible with water in every proportion.

Acetic acid comes into commerce in the most varied degrees of purity. The principal impurities are hydrochloric and sulphuric acid (especially harmful in printing), iron, lead, lime, sulphurous acid and empyreumatic substances. The strength is estimated by titration: 50 grammes are diluted to 1,000 c.c. and 50 c.c. of the solution titrated with N caustic soda and phenolphthaleïn, 1 c.c. of N soda = 0·06 gramme of anhydrous acetic acid. Crude, highly coloured acid frequently cannot be titrated directly; in this case litmus paper must be used as indicator or Mohr's process employed. Mohr treats 5 grammes of acetic acid at the boiling point with a weighed excess of calcium carbonate, filters, washes the residue with hot water and estimates the undecomposed carbonate by titration (excess of nitric acid titrated back by N soda), from which the proportion of acetic acid can be calculated. In addition the residue on evaporation at 110°C. may be estimated; very little should remain. If free sulphuric acid is present, it can be extracted by alcohol from the evaporated residue (sulphates are precipitated), the solution filtered, diluted and titrated with $\frac{1}{10}$ N soda or estimated gravimetrically by means of barium chloride.

Application.—Added to the printing colour, in the proportion of about 50 grammes of acetic acid of 4°B. to 1 kilo., in order to prevent the formation of lakes before printing; in dyeing with dyes sensitive to acids (*e.g.* eosine); for brightening silk; in wool-dyeing to correct calcareous waters; for thick woollen materials to secure more equal dyeing and better dyeing throughout (also as ammonium salt); as an addition to ferric acetate.

Specific Gravity of Acetic Acid at 15°*C.* (*Oudemans*).

Specific gravity.	Acetic acid per cent.	Specific gravity.	Acetic acid per cent.	Specific gravity.	Acetic acid per cent.	Specific gravity.	Acetic acid per cent.
0·9992	0	1·0363	26	1·0631	52	1·0748	77
1·0007	1	1·0375	27	1·0638	53	1·0748	78
1·0022	2	1·0388	28	1·0646	54	1·0748	79
1·0037	3	1·0400	29	1·0653	55	1·0748	80
1·0052	4	1·0412	30	1·0660	56	1·0747	81
1·0067	5	1·0424	31	1·0666	57	1·0746	82
1·0083	6	1·0436	32	1·0673	58	1·0744	83
1·0098	7	1·0447	33	1·0679	59	1·0742	84
1·0113	8	1·0459	34	1·0685	60	1·0739	85
1·0127	9	1·0470	35	1·0691	61	1·0736	86
1·0142	10	1·0481	36	1·0697	62	1·0731	87
1·0157	11	1·0492	37	1·0702	63	1·0726	88
1·0171	12	1·0502	38	1·0707	64	1·0720	89
1·0185	13	1·0513	39	1·0712	65	1·0713	90
1·0200	14	1·0523	40	1·0717	66	1·0705	91
1·0214	15	1·0533	41	1·0721	67	1·0696	92
1·0228	16	1·0543	42	1·0725	68	1·0686	93
1·0242	17	1·0552	43	1·0729	69	1·0674	94
1·0256	18	1·0562	44	1·0733	70	1·0660	95
1·0270	19	1·0571	45	1·0737	71	1·0644	96
1·0284	20	1·0580	46	1·0740	72	1·0625	97
1·0298	21	1·0589	47	1·0742	73	1·0604	98
1·0311	22	1·0598	48	1·0744	74	1·0580	99
1·0324	23	1·0607	49	1·0746	75	1·0553	100
1·0337	24	1·0615	50	1·0747	76		
1·0350	25	1·0623	51				

Note.—The specific gravities above 1·0553 correspond to *two* solutions of very different strengths. In order to ascertain whether the acid under examination has a greater strength than acid of the maximum density (78 per cent.), a little water is added; if the specific gravity is increased, the acid was stronger than 78 per cent.; if the specific gravity is decreased, the acid was weaker than 78 per cent.

Acetates are tested for the ordinary impurities: sulphate, chloride, iron, lead, lime, etc. An estimation of acetic acid is generally unnecessary; it is simpler to estimate the metal, as, *e.g.*, in chromium acetate. It is necessary also to examine for excess of free acid or mineral acid (basicity) and complete solubility. If it is necessary to estimate acetic acid, a weighed quantity of the acetate is distilled with phosphoric acid and the distillate titrated. For example, 5 grammes of acetate of lime are distilled almost to dryness with 50 c.c. of water and 50 c.c. of phosphoric acid (specific gravity 1·2) from a retort provided with a Liebig's condenser; the contents of the retort are diluted with 50 c.c. of water and again distilled; a third distillation is similarly performed. In the distillate, or in an aliquot portion, the acetic acid is estimated by N caustic soda, 1 c.c. = 0·06 gramme of acetic acid = 0·079 gramme of anhydrous calcium acetate (Fresenius, *Quant. Anal.*, ii., 201). The acetates or their solutions are frequently made by the consumer, in which case the materials must be tested.

Sodium acetate is used in calico-printing and wool-dyeing.

Calcium acetate in printing alizarine red; an important raw material for various preparations of acetic acid. It should be 90 per cent. pure.

Lead acetate, sugar of lead, contains as impurities calcium acetate, lime, lead carbonate; is used for chrome yellow and orange; hardly ever used now for weighting raw silk.

Aluminium Acetate, "Red Mordant".—In Turkey red dyeing (1° to 8°B.) in a similar manner to aluminium sulphocyanide and sulphate; for fixing wood colours in printing; for mordanting silk; for softening chappe (see footnote, p. 84); for water-proofing.

Tin acetate is used to discharge azo-dyestuffs in calico-printing.

Chromium acetate in printing alizarine colours (generally chromous acetate) instead of pyrolignite of iron.

Copper acetate (normal copper acetate or verdigris = $Cu(C_2H_3O_2)_2.H_2O$) is used in blue printing and in calico-printing to fix logwood. "French verdigris" is the basic salt $Cu_2(C_2H_3O_2)_2(OH)_2$. The percentage of copper is estimated as in copper sulphate, the basicity as in ferric sulphate, the acetic acid by distillation with phosphoric acid, as in other acetates (see above).

Ferrous Acetate, Acetate or Pyrolignite of Iron, Black Mordant.—Mineral acid should be absent, but the preparation generally contains free acetic acid; it frequently contains ferric chloride, Glauber's salt, sodium chloride and tarry matter, and is adulterated with ferrous sulphate. *Pure* ferrous acetate is obtained by double decomposition from lead acetate and ferrous sulphate (used for chamois and hence known as "chamois mordant"). *Pyrolignite* of iron is prepared from ferrous sulphate and crude acetate (pyrolignite) of lime, thus it generally contains some sulphate. It also contains many tar products in solution, such as pyrocatechol, phenols, acetone, wood-spirit, etc., but it should contain nothing insoluble. This is a very important point. A mordant, which deposits tar constituents on standing in the air, especially matter of a soft, sticky and resinous nature, must be rejected, since its use would involve the risk of producing spotted pieces. The deposition of a light brittle crust is, on the other hand, often not to be avoided and is without danger. The strength of pyrolignite of iron varies greatly, and must accordingly be estimated, together with the sulphuric acid. The following are the results of analyses :—

I.	1·6	per cent.	of ferrous	acetate,	7·8	per cent.	of SO_3
II.	4	,,	,,	,,	5·0	,,	,,
III.	5·3	,,	,,	,,	traces		,,

In case free acetic acid and, except sulphuric, no other acid is present, the acetate of iron may be estimated by difference.

Total Iron.—Since the tarry matters interfere with the estimation, an accurately weighed quantity is evaporated to dryness, the residue ignited and the iron determined in the ash gravimetrically or volumetrically (see *Ferric Sulphate*).

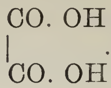
Sulphuric acid is similarly estimated by evaporation of a weighed quantity, ignition of the residue with soda or soda and saltpetre, solution in dilute hydrochloric acid and precipitation with barium chloride as barium sulphate.

In case no noteworthy quantity of alkali salts or of other metals except iron is present, the sulphuric acid is calculated as ferrous sulphate and the remainder of the iron taken as acetate.

Acetic Acid.—If noteworthy quantities of alkali salts are present (evaporation of the filtrate after precipitation of the iron by ammonia), such a method of calculation cannot be adopted. The acetic acid is then estimated by distillation with phosphoric acid and titration of the distillate (see *Acetates*).

Practical Test.—In addition to the chemical analysis a practical test must be made, since it is impossible to calculate the effect of the tarry matters. This test will naturally approach as nearly as possible to the large scale practice.

Application.—In silk-dyeing (blue-black); for dark shades of alizarine red; in cotton-dyeing more rarely on account of its high price. "Chamois mordant" (above) is used for pale chamois shades.

Oxalic Acid (and **Oxalates**), 
$\begin{array}{l} CO.\,OH \\ | \\ CO.\,OH \end{array} .\ 2H_2O = 126$; solubility = 1 : 9.

Oxalic acid and its salts often contain sulphuric acid; if this be absent, an acidimetric estimation is sufficient in the case of oxalic acid.

Total Acid.—50 grammes are dissolved to 1,000 c.c. and 50 c.c. titrated with N caustic soda and phenolphthaleïn; 1 c.c. of N soda = 0·063 gramme of crystallised oxalic acid. If sulphuric acid is present in quantity, the

Oxalic acid is estimated by oxidation with permanganate. To 20 c.c. of the above solution are added 25 c.c. of sulphuric acid (1 : 3) and the mixture titrated at 60° to 70°C. by $\frac{1}{5}$ N permanganate. 5 mols. of oxalic acid = 2 mols. of $KMnO_4$, or 1 c.c. of $\frac{1}{5}$ N permanganate solution = 0·0126 gramme of $C_2H_2O_4 .\ 2H_2O$.

A quantitative estimation of *sulphuric acid* is generally unnecessary; if required, the sulphuric acid is precipitated as barium sulphate, etc. Otherwise, the difference between total acid and oxalic acid gives the sulphuric acid.

It should be observed that oxalates are not included in an acidimetric estimation, but are titrated together with oxalic acid by permanganate; also sulphates are included in a gravimetric estimation of SO_3, but not determined by titration of the total acid.

In other respects oxalic acid must give a clear solution and contain little lime.

The amount of oxalic acid in the *oxalates* is estimated as above with permanganate, the metal according to the ordinary processes.

Application.—In dyeing wool with cochineal (formerly tin crystals were alone used); in printing on wool instead of acetic acid (20 grammes of oxalic acid per 1 kilo. of colour);

in mordanting wool with bichromate, etc. (in place of tartar); ammonium oxalate is used—similarly to ammonium acetate—in order to make the fibre absorb the dye (induline) more slowly and evenly; in calico-printing to remove iron and alumina; in washing to remove iron moulds; to remove Prussian blue from the fibre; as an addition to direct black.

Chromium oxalate in calico-printing.

Stannic oxalate in printing alizarine colours.

Stannous oxalate as a reducing agent and discharge, especially for silk and half-silk goods.

Ammonium oxalate has been proposed as a substitute for tartar emetic, but has been little used (see *Tartar Emetic*).

Tartaric acid, $C_2H_2(OH)_2(CO.OH)_2 = 150$; readily soluble in water.

The ordinary impurities of tartaric acid are sulphuric acid, oxalic acid and lime (detected by barium chloride, calcium sulphate and ammonium oxalate respectively). The residue on ignition, the solubility, and the amount of acid may also be examined; the last-named is estimated by titration with standard soda or baryta water. 1 c.c. of N soda = 0·075 gramme of tartaric acid.

If the tartaric acid is very crude, and largely contaminated by other acids or deep in colour, it may be examined by the method II. given for tartar (below).

Application.—Tartaric acid, on account of its high price, is as little used as possible, but is still extensively employed. In silk-dyeing for brightening; in calico-printing brighter shades are produced by tartaric acid (5 to 10 grammes per kilo. of colour); in wool-dyeing. It is, however, most largely employed in the form of tartar.

Potassium bitartrate, tartar, $C_2H_2(OH)_2(CO.OH)(CO.OK) = 188$; solubility, cold water = 0·4 : 100; hot water = 71 : 100.

It comes into the market in very varying degrees of purity: "crude," "half crystal," and "pure"; according to the purity and colour one or the other method of examination must be used.

I. Crude tartar: 10 grammes of tartar are boiled with 7 grammes of potassium carbonate and 150 c.c. of water for twenty to thirty minutes in a 200 c.c. flask, the volume made up to the mark, the flask shaken, 100 c.c. filtered, evaporated to 25 c.c., 5 c.c. of acetic acid added, the mixture stirred, covered, and warmed for fifteen minutes on the water-bath. Then 100 c.c. of absolute alcohol are added, the liquid well stirred and allowed to stand for fifteen minutes. The separated pure tartar is filtered off with the pump, and washed with 96 per cent. alcohol, until the alcohol after dilution with water gives an alkaline reaction with one drop of N caustic soda. The tartar is then titrated by $\frac{1}{2}$ N caustic soda, which has been standardised against tartaric acid or tartar, using sensitive litmus paper or phenolphthaleïn as indicator (see Fresenius, *Zeits. anal. Chem.*, xxii., 270).

II. Scheurer-Kestner's process is simpler (*Comptes rend.*, lxxxvi., 1024). The tartar is extracted with hydrochloric acid, the filtrate neutralised with caustic soda and the tartaric acid precipitated by means of calcium chloride as calcium tartrate. The precipitate is washed, dried and calcined; in the residue the calcium carbonate is determined volumetrically or gravimetrically and the result calculated to tartar. 1 mol. of potassium bitartrate = 1 mol. of $CaCO_3$.

III. If the tartrate is pure and free from foreign acids (or other substances combining with alkali) it can be titrated directly with N soda in a similar manner to tartaric acid; 2 to 3 grammes are taken. 1 c.c. of N soda = 0·188 gramme of tartar.

Application.—Principally in combination with potassium bichromate in mordanting wool; recently tartar has been subjected to an energetic competition by cheaper substitutes.

Most of the substances offered as substitutes for tartar consist of oxalic acid, common salt, alum, Glauber's salt and similar substances. Herzinger has published the following recipe: 2 parts of common salt, 2 parts of tartaric acid and 5 parts of Glauber's salt are mixed and allowed to stand for some time, then 4 litres of water, 200 grammes of tin crystals and 400 grammes of sulphuric acid are added ("Tartar preparation" = sodium bisulphate, which see).

Tartar Emetic, Antimonyl Potassium Tartrate, $C_4H_4O_6K(SbO).\frac{1}{2}H_2O = 332$; solubility, cold water = 7 : 100; hot water = 53 : 100. Contains 43·4 per cent. of antimony oxide.

The antimony and tartaric acid are determined. The principal impurity is oxalic acid, which is detected in the filtrate from the antimony, after driving off the sulphuretted hydrogen, by means of ammonia and calcium chloride or sulphate.

Antimony.—(*a*) 10 grammes of the substance are dissolved to 1,000 c.c. and in 50 c.c. (= 0·5 gramme of substance) the antimony is precipitated by sulphuretted hydrogen, brought on to a weighed filter paper (dried at 100° C.), the precipitate dried at 100° C. and weighed as Sb_2S_3.

(*b*) 50 c.c. of the above solution are mixed with 20 c.c. of a 10 per cent. sodium bicarbonate solution and 25 c.c. of a 2 per cent. bleaching powder solution and the mixture well stirred. If sufficient bleaching powder has been added, a drop of the solution gives a blue coloration with potassium iodide starch paper. It is now titrated with $\frac{1}{10}$ N sodium arsenite solution, until a drop of the solution no longer gives a blue coloration when brought upon potassium iodide starch paper. A volume of bleaching powder solution, equal to that used above, is now titrated alone and the chlorine required to oxidise the antimony calculated to arsenite (W. B. Hart). 1 c.c. of $\frac{1}{10}$ N sodium arsenite solution = 0·006 gramme of Sb = 0·0072 gramme of Sb_2O_3.

Example: 0·5 gramme of tartar emetic, 25 c.c. of bleaching powder solution, and 7·5 c.c. of $\frac{1}{10}$ N arsenite solution (25 c.c. of bleaching powder solution require 36·5 c.c. of arsenite); 36·5 – 7·5 = 29 c.c. of arsenite solution are used by the antimony, then

$$\frac{0{\cdot}0072 \times 29 \times 100}{0{\cdot}5} = 41{\cdot}76 \text{ per cent. of } Sb_2O_3.$$

Tartaric Acid.—The filtrate from the antimony sulphide is boiled to drive off sulphuretted hydrogen, and the tartaric acid determined by one of the methods described under *Tartar*, *e.g.*, method II. It should be stated that the percentage of antimony alone does not indicate the purity of the compound, since tartar emetic may be adulterated by other antimony salts, containing even more antimony than tartar emetic (antimony salt, $SbF_3.(NH_4)_2SO_4$, contains 47 per cent. of Sb_2O_3; double antimony fluoride, $SbF_3.NaF$, contains antimony equivalent to 66 per cent. of Sb_2O_3).

Application.—As a mordant for cotton in combination with tannin, in fixing basic dyes; antimony potassium oxalate, $K_3Sb(C_2O_4)_3.6H_2O$ (23·7 per cent. of Sb_2O_3) has been introduced for the same purpose, but cannot replace tartar emetic in all cases. Another substitute is "antimony salt" or "tartar emetic substitute" (see above). Tartar emetic is also one of the materials from which the little-used aniline tartrate is prepared.

Citric Acid, $C_3H_4(OH)(CO.OH)_3.H_2O$ = 210; readily soluble in water.

Generally applied in the form of lemon juice, which occurs in commerce as a 25 per cent. syrup. Often adulterated by cheaper acids—oxalic, tartaric and sulphuric. Heavy metals may also be found. Oxalic acid is detected by means of lime water and ammonia, or calcium sulphate or chloride. Tartaric acid is detected by heating for one hour with strong sulphuric acid at 60° to 70° C.; if tartaric acid be present, a black or brown coloration is obtained, 0·5 per cent. may readily be detected in this manner. The citric acid, or lemon juice, is also titrated: 50 grammes are dissolved to 1,000 c.c. and 100 c.c. titrated with N caustic soda and phenolphthaleïn, 1 c.c. of N caustic soda = 0·07 gramme of crystallised acid = 0·064 gramme of anhydrous citric acid.

Application.—As a consequence of the high price is little used. Principally in silk-dyeing for brightening (lemon juice); also as a discharge for alizarine colours; then, similarly to oxalic acid, to remove alumina and iron from the fibre; in finishing silk goods.

Lactic Acid, $CH_2(OH).CH_2.CO.OH = 90$. Miscible with water.

Quite recently lactic acid has been brought into the market, as a substitute for tartar, oxalic acid and tartar preparations, in the form of a 50 per cent. syrup. The strength is determined, as of the other organic acids, with N caustic soda and phenolphthaleïn. 1 c.c. of N soda = 0·09 gramme of lactic acid. The impurities are sulphuric, acetic and oxalic acids. If these are present in appreciable quantity a quantitative estimation, *not* acidimetric, must be made under proper conditions. If oxalic acid be present, its percentage must be deducted.

Lactic Acid.—1 gramme of the acid is dissolved in 100 c.c. of water, 3 grammes of caustic potash (dissolved in a little water) added, and a 5 per cent. permanganate solution added, with continuous shaking, until the originally green solution becomes bluish-black, which colour must remain in the subsequent boiling. During boiling manganese peroxide is precipitated. After cooling, hydrogen peroxide is added until the solution is colourless, when the liquid is again boiled. The precipitate is filtered off, washed with hot water and the oxalic acid estimated in the filtrate by ordinary methods. The reaction is $C_3H_6O_3 + 5O = CO_2 + C_2H_2O_4$

$+ 2H_2O$ (Ulzer and Seidel, *Monatsh. für Chem.*, 1897, 138).

Application.—Principally as an assistant in mordanting wool with potassium bichromate; similarly with sulphuric acid and bichromate; the acid potassium salt (lactolin) is also recommended. Said to give better equalisation than tartar. Proposed for brightening silk. Put on the market as zinc, etc., salts by the firm of Böhringer.

CYANOGEN COMPOUNDS.

Sulphocyanides, CNS – M, where M indicates a metal. The sulphocyanic acid may be estimated volumetrically in nitric acid solution with silver nitrate (the reverse of the silver or chlorine titration by Volhard's method). As in the titration of copper (which see), it is important to add the silver nitrate in excess, *then* acidify with nitric acid and titrate the excess of silver with $\frac{1}{10}$ N ammonium sulphocyanide solution; 1 mol. of alkaline sulphocyanide = 1 mol. of $AgNO_3$ or 1 c.c. of $\frac{1}{10}$ N silver nitrate solution = 0·0097 gramme of KCNS.

Neutral salts may be titrated directly with silver solution, using potassium chromate as indicator.

Application.—The sulphocyanides find very restricted employment in calico-printing; in dyeing they are not used.

Aluminium sulphocyanide for alizarine and as a resist for aniline black.

Tin sulphocyanide for alizarines.

Chromium sulphocyanide, instead of chromium acetate, for silk and half-silk goods.

Potassium sulphocyanide, on account of the ease with which it is decomposed, is used as a resist and discharge. It is printed with a suitable thickener.

Barium sulphocyanide has only been used experimentally.

Cuprous Sulphocyanide is used in a very restricted manner for the sake of the copper it contains.

Potassium Ferrocyanide, Yellow Prussiate of Potash; Sodium Ferrocyanide. $K_4Fe(CN)_6.3H_2O = 422$; solubility, cold water = 28 : 100; hot water = 100 : 100. $Na_4Fe(CN)_6.10H_2O = 484$; very soluble.

Potassium ferrocyanide and the less-used sodium salt may contain as impurities: potassium sulphate, carbonate and chloride; the potassium salt may contain the sodium salt. These impurities are detected in the solution immediately or after fusing in a porcelain crucible with potassium nitrate and extracting with water (Cl). The potassium or sodium ferrocyanide is directly estimated by the following processes:—

I. 20 grammes are dissolved in 1,000 c.c.; 20 c.c. of the solution, mixed with 250 c.c. of water and 20 c.c. of sulphuric acid (1 : 3), are titrated with $\frac{1}{5}$ N permanganate, until the red coloration appears. 1 c.c. of $\frac{1}{5}$ N permanganate = 0·0112 gramme of Fe = 0·0845 gramme of potassium ferrocyanide (Fe × 7·546 = potassium ferrocyanide). The permanganate solution may be standardised against iron or against pure potassium ferrocyanide (20 grammes in 1,000 c.c., 10 c.c. titrated). For example, if 50 c.c. of permanganate are required, then 1 c.c. of permanganate = 0·004 gramme of ferrocyanide = 2 per cent; each 0·5 c.c. of permanganate = 1 per cent. of ferrocyanide. The above degree of dilution must be maintained; in more concentrated solutions the end-point is not so sharp. Gintl (*Zeits. anal. Chem.*, vi., 446) adds a trace of ferric chloride as indicator and titrates until the blue coloration disappears (see also Fresenius, *Quant. Anal.*, i., 378).

II. In case potassium carbonate is absent, potassium ferrocyanide may be titrated by a standard zinc sulphate solution in an ammoniacal tartaric acid solution (reversed titration of zinc salts, see also Donath and Hattensauer, *Chem. Zeit.*, 1890, 323; *J. S. C. I.*, 1890, 554).

1 mol. of $K_4\ Fe(CN)_6$ = 2 mols. of zinc salt = 2 atoms of zinc.
1 gramme of $K_4\ Fe(CN)_6.\ 3H_2O$ = 0·3074 gramme of Zn.

The zinc is entirely precipitated when a drop of the solution begins to give a blue coloration with a mixture of ferric chloride and acetic acid. Accordingly, the potassium ferrocyanide solution must be run into the solution of the zinc salt, or at the commencement a little ferric chloride, which remains unprecipitated by the ferrocyanide, is added to the zinc solution (zinc sulphate is best).

Application.—Principally for the preparation of Prussian blue upon cotton, wool and silk, especially for silk which is to be dyed black; also for aniline black (very restricted use).

Potassium Ferricyanide, Red Prussiate of Potash, $K_6Fe_2(CN)_{12}$ = 658; solubility, cold water = 40 : 100; hot water = 80 : 100.

The principal impurities are potassium sulphate and ferrocyanide. The sulphate is estimated as in potassium ferrocyanide; the ferrocyanide may be determined by direct titration with permanganate, which may in this case conveniently be $\frac{1}{25}$ N. The ferricyanide is estimated indirectly by titration with permanganate after reduction to ferrocyanide. The reduction is accomplished, according to Gintl (Mohr, *Titrirmethoden*, 6 Aufl., 237), very easily by means of sodium amalgam, several lumps of which, the size of a pea, are brought into a neutral or alkaline solution. The reduction is complete in ten minutes, the solution is then titrated with $\frac{1}{5}$ N permanganate, 1 c.c. of which = 0·0112 gramme of Fe = 0·1318 gramme of potassium ferricyanide.

Application.—As an oxidising agent for steam aniline black; as a discharge for indigo, alizarine blue, cœruleïn, etc. (MgO or water glass is then added as a diluent); it is occasionally used for logwood black to oxidise the colouring matter.

Derivatives of the Fats.

Soap.—Soaps consist principally of the neutral sodium or potassium salts of the fatty acids and water, together with larger or smaller quantities of common salt, caustic and carbonated alkalis, unsaponified fat, glycerin, etc., as impurities. The smaller the quantity of these impurities the higher is the quality of the soap.

Soda soaps, which are "salted out" in the process of manufacture, are known as curd soaps; if they contain added water they are called "cut" soaps, and if they contain the constituents of the residual liquors of the manufacture, they are known as "filled" soaps, which thus contain larger quantities of glycerin, caustic soda, salt, etc.

Potash soaps cannot be "salted out" with common salt, because a transformation into soda soap and potassium chloride would occur, they therefore contain, similarly to the "filled" soaps, all the impurities contained in the residual lyes—glycerin, excess of caustic potash, etc. On account of their consistency they are known as soft soaps.

In addition to these pure soaps, reduced qualities also come into the market, to which quantities of various materials have been added, *e.g.* water glass, chalk, starch, borax, kieselguhr, Glauber's salt, mineral oil, clay, barytes, etc. Such soap is tabooed in the textile industries; it is chiefly used for toilet purposes. The impurities are most readily detected by dissolving the soap in absolute alcohol, or in the ash. Pure soaps alone will be considered here.

The composition of soaps varies greatly; for each special purpose special properties are required. Calvert gives the following as the compositions of normal soaps for various consumers.

	Fatty acid per cent.	Soda per cent.	Water per cent.
Normal soap for Calico printers . .	64·0	6·0	30
,, ,, ,, Cotton dyers . .	66·0	7·0	27
,, ,, ,, Silk dyers . . .	61·9	8·1	30
,, ,, ,, Woollen manufacture	61·4	8·6	30
Best soap for madder violet . . .	60·4	5·6	34

The cause of the varying composition of soaps is to be found in the use, in their manufacture, of the most varied oils and fats, which have the power of combining different amount of alkali and water. There are used : olive oil from various sources, the fatty acids of olive oil, palm oil, cocoanut oil, tallow, hemp-seed, rape seed and linseed oils, etc. Assuming that a soap contains no artificial additions, the following are the most important characters : the proportion of fatty acids, total alkali, free alkali and unsaponified fat. Of the greatest importance are also the nature and quality of the fatty acids ; the proportion of common salt is less important. The percentage of water, which may be obtained by difference, may also conveniently be directly determined (R. Benedikt, *Analyse der Fette und Wachsarten ;* Lewkowitsch, *Analysis of Oils, Fats and Waxes ;* C. Schädler, *Untersuchung der Fette, Oele und Wachsarten*).

The most speedy and accurate method of conducting a soap analysis is commenced by making a stock solution, in which as many constituents as possible are estimated.

Analysis.—20 to 25 grammes of soap, cut from the interior of the piece, are dissolved to 1,000 c.c.

Water.—50 c.c. of the solution are placed in a porcelain dish containing about 10 grammes of ignited quartz sand and a small glass spatula, which have been weighed with the dish. The contents are evaporated to dryness on the water-bath and dried at 100° C., with the addition of a little alcohol, to constant weight. The addition of alcohol (a few c.c.) considerably facilitates the evaporation of the last portions of water. The loss in weight is water. This method is more rapid and accurate than the usual process of drying soap shavings first at 50° to 70°C. and then at 100°C. The latter process generally gives too little water, which remains enclosed in the interior of the soap.

Fatty Acids.—(*a*) 100 c.c. of the solution are decomposed with 20 c.c. of normal sulphuric acid in a thin-walled beaker, which has been weighed together with a glass rod. The mixture is heated on the water-bath until the fatty acids have separated as a clear layer and the lower aqueous solution has become almost completely transparent. The hot liquid is then filtered through a good Swedish filter paper (previously dried at 100°C. and weighed), and the oily layer washed with hot water until the filtrate no longer shows an acid reaction to sensitive neutral litmus paper; 200 c.c. of hot distilled water are, as a rule, sufficient. The filtrate is retained for the estimation of total alkali.

The filter paper containing the fatty acids is then placed in the weighed beaker used for the decomposition, the funnel washed with a few drops of petroleum ether if it shows traces of fat, and the beaker and its contents dried at 100°C. to constant weight. The increase in weight gives the fatty acid, which is converted into the corresponding amount of fatty acid anhydride by deducting 3·25 per cent. The filtrate must be completely clear and free from every trace or globule of fat; an opposite condition may be ascribed to the use of an unsuitable paper, to incomplete moistening of the paper before filtering and to the "creeping" of melted acids along the folds of the paper and so over the edge.

Instead of a weighed filter, Gawalovsky (*Zeits. anal. Chem.*, xxiv., 219; *J. S. C. I.*, 1885, 369), prefers an ordinary unweighed paper, and washes the fatty acids, after they have solidified, with petroleum ether into the beaker. If the acids or the paper are wet, they are first moistened with a few c.c. of alcohol, which removes the water; the acids are then washed through with petroleum ether. The filter paper must be washed with petroleum ether until there is no trace of fat in the filtrate, *i.e.*, until about ten drops leave no residue on evaporation on a watch glass. Gawalovsky states that, when the fatty acids are dried together with the paper, capillary decompositions occur, which, however, according to the author's experience, hardly exceed 0·05 to 0·1 per cent. with careful working. On the other hand the process in which the acids are dried together with the paper, is far simpler and more rapid.

(*b*) Wax method. Instead of filtering off the fatty acids separated by sulphuric acid, they may be melted to a cake with about 10 grammes of pure wax (dry and not capable of giving up anything to water). The beaker is then rapidly cooled in ice or cold water, so that the cake of wax may separate completely from the glass. The cake is washed with cold water, freed from adhering drops of water by means of filter paper and dried in an exsiccator over sulphuric acid. The operation must be so conducted that the cake of wax (or paraffin) separates entirely, without leaving traces of wax or fat on the glass; with some practice this is easy. If the separation is not successfully accomplished, the beaker may be dried and weighed, and the increase in weight added to the weight of the cake. This method generally gives appreciably higher results than the preceding, since more or less water always remains enclosed in the interior of the cake and cannot be removed even by very long drying in the exsiccator.

Total Alkali.—(*a*) The filtrate from the fatty acids (*a*) or the solution obtained after removing and washing the wax cake (*b*) is titrated back with normal caustic soda and phenolphthaleïn. The volume of soda solution used deducted from the 20 c.c. of normal acid originally added, corresponds to

100 c.c. of the soap solution. 1 c.c. of normal sulphuric acid $= 0{\cdot}031$ gramme of Na_2O.

(*b*) 100 c.c. of the soap solution may also be directly titrated with normal acid and methyl orange until the pink coloration appears. The results agree very well with those obtained by process (*a*). 1 c.c. of normal acid $= 0{\cdot}031$ gramme of Na_2O.

Sodium Chloride.—The solution used for the estimation of the total alkali may now serve for the determination of sodium chloride. In case the solution is coloured pink, it is decolorised by 1 drop of $\frac{1}{10}$ N sulphuric acid or by means of the carbonic acid in the breath, a few drops of a neutral solution of potassium chromate are added and the liquid then titrated with $\frac{1}{10}$ N silver nitrate solution until the brown coloration begins to appear. 1 c.c. of $\frac{1}{10}$ N silver nitrate solution $= 0{\cdot}00584$ gramme of NaCl. Good curd soap generally contains 0·4 to 0·8 per cent. of NaCl.

Free Alkali.—The estimation of the free alkali is a delicate process, since only hundredths of a per cent. have frequently to be determined and only 0·1 per cent. is often quite impermissible. An approximate idea of the alkalinity of a soap may be obtained by moistening a freshly cut surface with mercuric chloride solution: a brownish-yellow to reddish-brown coloration appears according to the amount of free alkali. The degree of alkalinity may also be roughly ascertained by the taste—by laying a freshly cut surface on the tongue.

Of the many chemical methods for the quantitative estimation of free caustic soda, the following is to be regarded as the most reliable and speedy:—

(*a*) 500 c.c. of the solution (10 to 12 grammes of soap) are heated with 200 grammes of common salt (neutral, or of known alkalinity, which is deducted in the calculation) in a flask, provided with a Bunsen valve, on the water-bath, until all the salt has dissolved, the soap has separated in curdy masses and the aqueous solution has become clear. The flask is then cooled, the contents filtered through a large folded filter into a 1 litre flask, the filter washed with a saturated solution of common salt, and the filtrate made up to one litre. Then 500 c.c. are brought into a beaker, excess of barium chloride solution is added, by which the carbonate is precipitated (see *Soda* and *Caustic Soda*), and the liquid titrated, without filtering, with $\frac{1}{10}$ N hydrochloric acid and methyl orange (not phenolphthaleïn) until the red coloration begins to appear. 1 c.c. of $\frac{1}{10}$ N hydrochloric acid = 0·0031 gramme of Na_2O as caustic soda. The second 500 c.c. are titrated with $\frac{1}{10}$ N hydrochloric acid and methyl orange, without the addition of barium chloride. 1 c.c. of $\frac{1}{10}$ N hydrochloric acid = 0·0031 gramme of Na_2O as soda + caustic soda. The difference between the two titrations gives the sodium carbonate.

In view of the small quantity of caustic soda which is generally to be determined, a correction of the following kind must inevitably be made: 200 grammes of common salt of the same quality are dissolved under exactly the same conditions, the solution filtered, brought to 1,000 c.c., and two quantities of 500 c.c. each titrated, with and without barium chloride, with $\frac{1}{10}$ N hydrochloric acid and methyl orange. The volumes of acid required in these experiments until the commencement of the red coloration (orange coloration) are deducted from the volumes used in the soap titrations. This consumption of acid, until the end-reaction occurs, is not to be ascribed alone to the alkalinity of the common salt; it would be necessary with perfectly neutral salt, since the sensitiveness of indicators in such dilute solutions and in strong salt solutions is less than in stronger solutions containing less salt. On the other hand a certain degree of dilution is necessary, since in strong solutions of soaps, the latter, on precipitation, enclose a certain amount of alkali, which would thus be lost. The use of phenolphthaleïn at these dilutions and in presence of so much common salt must be regarded as quite excluded and unreliable.

In the case of an almost neutral soap a quantity of 10 to 12 grammes is not sufficient; it is necessary to take a larger portion. Frequently 30 to 50 grammes of soap are dissolved in 500 c.c. of water, the soap precipitated as above with 200 grammes of salt, the liquid cooled, filtered into a 500 c.c. flask, and two quantities of 250 c.c. taken, etc. Exactly as described above, one half is titrated with $\frac{1}{10}$ N acid with, the other half without, barium chloride.

(*b*) 7 to 8 grammes of soap are dissolved to a very strong solution in 40 to 50 c.c. of water, salted out with common salt, the mixture filtered, the filtrate evaporated, the residue extracted with absolute alcohol, when alkaline carbonate remains undissolved, the solution diluted with water and titrated with $\frac{1}{10}$ N hydrochloric acid. This method, which is still much used, is to be regarded as quite untrustworthy since (1) caustic soda in appreciable quantity remains enclosed in soap separated from a strong solution; (2) during the evaporation of the solution the caustic soda has too much opportunity for absorbing carbonic acid from the air; and (3) the alcohol considerably diminishes the delicacy of the end-reaction.

(*c*) About 30 grammes of soap are dissolved in alcohol free from acid and the solution filtered; the filtrate is titrated with $\frac{1}{10}$ N hydrochloric acid and phenolphthaleïn. This method, also, is inaccurate, since (1) phenolphthaleïn is not sensitive in alcoholic solution; (2) phenolphthaleïn also always absorbs some alkali. Most *alcoholic* solutions of soaps do not give alkaline reactions with phenolphthaleïn even when they contain 0·1 per cent. of free soda (see also

C. and N. Draper, *Chem. News*, lv., 133; *Zeits. anal. Chem.*, 1886, 54; Gawalovsky, *Zeits. anal. Chem.*, 1888, 155: *J. S. C. I.*, 1885, 169; Hope, *Chem. News*, xliii., 219).

(*d*) The process of Moffit is quite inaccurate and inconvenient: 10 grammes of soap are dissolved in 150 to 180 c.c. of alcohol, the solution is filtered and the residue washed with alcohol in a hot-water funnel. A current of well-washed carbon dioxide is passed over the surface of the liquid, when the alkali is precipitated as carbonate, which is filtered off, washed with alcohol, dissolved in water and titrated in the usual manner (see also W. Waltke, *Chem. Zeit.*, 1896, 16, 137).

Alkaline carbonate is frequently not estimated, since, even in cases where the absence of caustic alkali is rigidly demanded, it is of little importance, as it can only advantageously neutralise the hardness of most waters. A considerable excess of soda would naturally be very harmful; the methods of soap-making, however, would almost always cause the excess of sodium carbonate to be in a certain proportion to the free caustic alkali. The soda may be determined as already described under *free alkali;* the filtrate from the salted-out cake of soap is titrated with $\frac{1}{10}$ N hydrochloric acid and methyl orange, with and without barium chloride. Each 1 c.c. of $\frac{1}{10}$ N acid of the difference = 0·0031 gramme of Na_2O as sodium carbonate. The harmfulness of the soda is, as already indicated, dependent upon the hardness of (proportion of lime in) the water. (For the estimation of sodium carbonate, silicate and borate in soap, see W. Waltke and Co., *Chem. Zeit.*, 1896, No. 3, p. 20.)

Glycerin may be estimated in the liquids which have been used for the determination of sodium chloride, total alkali or caustic alkali. The whole, or an aliquot portion, is evaporated to dryness, the residue extracted with absolute alcohol at 80° C., and the solution evaporated. When the glycerin obtained is impure, it can be titrated by Benedikt and Zsigmondy's method with permanganate in alkaline solution (see Benedikt, or Lewkowitsch, *Analysis of Oils*, etc).

$$C_3H_5(OH)_3 + 3O_2 = C_2H_2O_4 + CO_2 + 3H_2O$$
$$1 \text{ mol. glycerin} = 2 \text{ mols. } KMnO_4$$

Or, and this is preferable if the quantity of glycerin be small, a larger quantity of soap is taken (20 to 25 grammes), dissolved in water, sulphuric acid added to acid reaction, the fatty acids removed by one of the above methods—with wax or by filtering, the filtrate neutralised with potassium carbonate, evaporated to dryness, the residue ground in a mortar and extracted with absolute alcohol. The mixture is filtered into a tared beaker, and heated to constant weight on the water bath, or better at 50° to 60° C. The residue is glycerin, the result may be checked by titrating with permanganate as above.

Unsaponified Fat.—About 25 to 30 grammes of very finely powdered soap (when the water is estimated, the dry soap powder may be weighed; otherwise the moist soap must first be dried) are extracted in a Soxhlet's extractor for three to four hours with petroleum ether. The ether is evaporated and the fat weighed. Since soap is also soluble in petroleum ether to a slight extent, the ethereal solution must be poured off from any soap which has separated in the flask, the latter washed out and a correction made for the solubility of soap in petroleum ether: 100 c.c. of petroleum ether dissolve 0·01 gramme of Marseilles soap.

Melting and Solidifying Points of the Fatty Acids.—Endeavours to recognise the nature of the fatty acids are generally restricted to determinations of their melting and solidifying points. More rarely the specific gravity, iodine value, etc., are taken (see Benedikt, *Analyse der Fette und Wachsarten, Abschnitt*, x., xi.; Lewkowitsch, *Analysis of Oils, Fats and Waxes*, second ed., p. 123 *et seq.*).

The fatty acids, separated by dilute sulphuric acid from a strong boiling solution of the soap (100 : 800) contained in a beaker, are allowed to cool and kept in the cold for at least twenty-four hours. The odour evolved during the decomposition of the soap should be observed, important conclusions may thus sometimes be drawn. After removing and drying the fatty acids in the exsiccator, the melting and solidifying points are taken. In the case of a homogeneous product these temperatures should lie near together.

(*a*) Bach (*Chem. Zeit.*, vii. 356,) fills the acids into a narrow thin-walled test-tube, allows them to solidify and then warms the tube in a beaker of water heated with a small flame. The mass is stirred gently with a thermometer and the point noted at which the whole becomes completely clear. This is the melting point. The temperature, at which flocks begin to collect round the thermometer on cooling, is the solidifying point.

(*b*) Bensemann (*Repert. der anal. Chem.*, iv., 165) determines the beginning and ending of the fusion in the following manner : A piece of glass tube is drawn out so that the narrower and wider portions are equal in length, the narrow end is then fused up ; two to three drops of the fat are brought into the tube immediately above the constriction and allowed to solidify (solidification effected by one drop of ether). The tube is then placed in an upright or slightly inclined position in a beaker filled with cold water, in which a thermometer is hung. The beaker is heated as slowly as possible, by means of a small flame, until the drop of fatty acids just begins to run down. The temperature observed at this moment is "the commencement of fusion". The temperature, at which the turbid drop, on further heating, becomes quite transparent, is "the end of fusion".

The melting point is determined by many different methods, which give varying results. There is the uncertainty whether the temperature at which the fat begins to be fluid, or that at which it becomes clear, is to be designated the melting point. In general the former is taken, *i.e.*, the temperature described by Bensemann as "the commencement of fusion".

(*c*) Other methods give as the melting point that temperature at which, *e.g.*, the fat when placed in a narrow tube, open at both ends and heated in water, begins to rise in the tube.

(*d*) According to another method hollow glass spheres are placed in a wide test-tube filled with the fatty acids, which are continuously stirred with a narrow thermometer. The melting point is the temperature at which the spheres begin to rise.

(*e*) Similarly the solidifying point is the temperature at which the hollow spheres, when pressed down by means of the thermometer, do not rise again. For this operation cooling with water is generally sufficient; the temperature of the water should be several degrees below the melting point.

Molecular Weight of the Fatty Acids.—Frequently the molecular weight (or the saponification value) of the fatty acids indicates the nature of the fat or oil employed: 5 grammes of completely dry fatty acids are dissolved in 60 to 70 c.c. of hot neutral alcohol and titrated with normal alcoholic potash and phenolphthaleïn. The weight of fatty acid taken, multiplied by 1,000 and divided by the number of c.c. of normal potash used, gives the molecular weight. Thus 5 grammes of fatty acid required 18 c.c. of N caustic potash: $\frac{5 \times 1000}{18} = 277{\cdot}7$, the molecular weight required, assuming the acid to be monobasic.

Resin.—(*a*) If the soap is free from potash salts and unsaponified fat, the dry soap may, according to Barfoed, be extracted directly with ether-alcohol, in which sodium salts of the fatty acids are insoluble.

(*b*) If the soap contains potash salts the resin is estimated in the separated fatty acids, together with which it is precipitated (Barfoed, *Zeits. anal. Chem.*, xiv., 29). The fatty acids are neutralised with normal (or stronger) caustic soda, dried at 100° C. to constant weight, and heated with absolute alcohol (10 c.c. to 1 gramme of substance) to 80° C., when the resin and a portion of the fatty acid soap dissolve. After cooling, evaporated alcohol is replaced and 5 volumes of pure ether added, when the fatty acid soap is completely precipitated. Ether is then added to bring up to a definite volume, the mixture shaken from time to time for several hours, allowed to stand twenty-four to forty-eight hours, an aliquot part of the clear liquid withdrawn, brought into a weighed dish and evaporated to constant weight at 100° C.

(*c*) 2 to 3 grammes of the mixture of resin and fatty acids are dissolved in 25 to 30 c.c. of absolute alcohol in a small flask, and dry hydrochloric acid gas passed in, whilst cooling. The fatty acids are then converted into ethyl esters, whilst the resin acids remain unaltered. When no more gas is absorbed and the esters have separated (in about forty-five minutes) the flask is allowed to stand thirty minutes longer; 100 to 125 c.c. of hot water are then added, and the mixture shaken with 50 to 75 c.c. of petroleum ether in the separating funnel. The resin is dissolved by the petroleum ether, the solution is first washed with water and then shaken with a solution of 0·5 gramme of caustic potash in 5 c.c. of alcohol and 50 c.c. of water, when the resin acids separate; they are weighed on a tared filter (J. Laudin, *Chem. Zeit.*, 1897, 25).

Inorganic and organic added matter may be detected in the ash and aqueous or alcoholic soap solutions respectively. The principal substances used for this purpose have already been mentioned.

Applications.—On the widest scale for all textile fibres.

In silk-dyeing for scouring, soaping and dyeing, especially in black silk dyeing (neutral); in calico-printing; in wool-washing together with ammonia; in dyeing wool with Alkali Blue; in fulling woollen goods (free caustic soda and fat must be absent); for brightening (free alkali may be harmful). On a large scale in the cotton industry, to remove grease, etc.

"Boiled-off Liquor" is obtained in silk-dyeing. Its quality depends upon the strength of the soap employed and the nature of the silk fibre. In the absence of "boiled-off liquor" Sartori recommends the following:—

1. 400 grammes of Marseilles soap are dissolved in 15 litres of boiling water and a solution of 100 grammes of size added. Finally 0·05 litre of olive oil is introduced in small quantities. The solution should be used cold (*Färberei-Muster-Zeit.*, 1888, 333).

2. Another recipe is: 25 grammes of Marseilles soap and 4 to 6 grammes of gelatine are dissolved in 1 litre of water.

3. Or 20 grammes of Marseilles soap with 2 grammes of gelatine and 1 gramme of common salt are dissolved in 1 litre of water.

Application.—In silk-dyeing, generally in a bath acidified with sulphuric or acetic acid (for eosine). Glauber's salt (which see) is also often added.

Turkey Red Oil.—Widely different substances are sold under the names of "Turkey red oil" or "red oil," they vary according to the nature of the oil employed and the method of treatment. Amongst others the following are used: castor, olive, tournant, cotton-seed, rape-seed and cocoa-nut oils, oleïc acid, and mixtures of these. The most valuable Turkey red oil is obtained from castor oil.

Opinions vary as to the nature of the reaction (see Benedikt, p. 147 *et seq.;* Lewkowitsch, second edition, p. 726). The greater portion of Turkey red oil consists of ricinoleïc acid, the smaller—but more valuable—part of ricinoleo-sulphuric acid and hydroxyoleïc acids. The Badische Anilin-und Sodafabrik accordingly bring two qualities into the market: Turkey red oil D = sodium ricinoleate, and Turkey red oil F = sodium (or ammonium) ricinoleo-sulphate. The method of preparation consists in acting upon the above-mentioned oils with fuming sulphuric acid and then neutralising with soda or ammonia.

Turkey red oil is analysed in a similar manner to soaps. Preliminary examination: The oil must be slightly alkaline or neutral; it must yield a complete emulsion with water, from which drops of oil separate only on long standing. These drops of oil must give a clear solution in ammonia, if they do not give a clear solution they contain unsaponified fat.

Water.—According to Stein, 10 grammes of the oil are melted together with 25 grammes of dry wax in about 75 c.c. of a saturated solution of common salt. The cake is dried and weighed. The increase in weight of the wax represents anhydrous oil; the difference between this and the weight taken gives the water.

Total Fat (Brühl, *Zeits. anal. Chem.*, xxi., 448, and Stein, *Ber.*, xii., 1,174).—4 grammes of the sample are weighed into a thin-walled hemispherical glass dish, which was previously weighed together with a glass rod, 20 c.c. of water are then gradually added with continuous stirring. If the liquid is turbid, ammonia is added until the reaction is slightly alkaline towards phenolphthaleïn, 15 c.c. of sulphuric acid (1 : 1) are then added, next 6 to 8 grammes of stearic acid. The liquid is then gently boiled, until the fat separates clear, when it is allowed to cool, the solidified cake removed together with the glass rod and placed on filter paper. The residual liquid is again heated in the dish until the particles of fat have united to 1 or 2 drops. The dish is removed from the water-bath, and inclined so that the drops of fat adhere to the side. The liquid is poured off, the dish washed out with water and the cake returned to it. The dish is then heated over a small flame, which must not reach the bottom, with continuous and uninterrupted stirring by means of the glass rod, until the crackling sound is no longer heard and white vapours begin to be evolved. The dish is then allowed to cool, weighed, and the weight of dish, glass rod and stearic acid deducted. The remainder gives the total fat.

Neutral Fat.—About 30 grammes of the sample are dissolved in 50 c.c. of water, 20 c.c. of ammonia and 30 c.c. of glycerin added and the mixture twice extracted with 100 c.c. of ether. The ether is shaken with water in order to remove traces of soap, the solvent distilled off, the residue brought into a tared glass dish, dried first on the water-bath, then in the air-bath, and weighed.

Soluble Sulphated Fatty Acids (sulphuric esters of the fats).—The value of Turkey red oil depends principally on the proportion of ricinoleo-sulphuric acid : 5 to 10 grammes of the sample are dissolved in 25 c.c. of water in a thick glass flask, 25 c.c. of fuming hydrochloric acid added and the mixture heated for one hour in the oil-bath at 130° to 150° C. After diluting and transferring to a beaker, the layer of oil is filtered off. This is most easily accomplished if an unweighed quantity of stearic acid be added, the liquid boiled and allowed to cool. In the filtrate the sulphuric acid is precipitated by barium chloride, the sulphuric acid present as sulphate (see below) deducted, and the remainder calculated into ricinoleo-sulphuric acid by multiplication by the factor 4·725.

Fatty acids may be present either free or combined with soda or ammonia. The amount is found by deducting the neutral fat and sulphated acids from the total fat (see above). The difference corresponds to the fatty acids, which, after deduction of 3·15 per cent., may be inserted as acid anhydrides in the result of the analysis.

Sulphuric Acid.—In order to estimate the sulphuric acid present as alkaline sulphates, the solution of the oil in ether is several times shaken with a few c.c. of a saturated solution of pure sodium chloride. The extracts are united, diluted, filtered and precipitated with barium chloride. The total sulphuric acid contained in the oil may also be estimated by fusing with caustic potash and saltpetre, and the proportion of sulphated acids or sulphates calculated by difference.

Ammonium and Sodium.—7 to 10 grammes of oil are dissolved in ether and shaken 4 times with quantities of 5 c.c. of dilute (1 : 6) sulphuric acid. The united acid extracts are evaporated on the water-bath for the estimation of soda, freed from excess of sulphuric acid by heating on the sand-bath and the residue finally converted by ignition into sodium sulphate, which is weighed (142 parts of Na_2SO_4 = 62 parts of Na_2O). In order to estimate ammonia, the oil is similarly extracted with dilute sulphuric acid, the extracts distilled with excess of caustic potash and the ammonia caught in a measured volume of standard sulphuric acid, the excess of which is titrated at the end of the operation (see also *Ammonium Salts*).

Nature of the Oil.—The acetyl and iodine values are taken of the total fat separated without the addition of stearic acid (see Benedikt). If pure castor oil was used in the manufacture of the Turkey red oil, the former value is 140 or higher, the latter 70 or rather lower. If one or both of these values are found much smaller, the Turkey red oil has been made from a mixture of castor oil with another oil or from another oil alone, it is then to be regarded as of little value (olive oil, cotton-seed oil, oleïc acid, etc., see above and Benedikt).

Practical Test.—The suitability of the oil should be examined by means of a practical test.

Application.—In dyeing and printing Turkey reds. In finishing silk, silk mixture and cotton goods.

TANNINS.

There is a large number of materials containing tannin (tannic acid, digallic acid) which are principally used on account of that constituent, and are evaluated accordingly: gall-nuts (30 to 40 per cent.); *Knoppern* (see p. 120); mirabolans, divi-divi (30 per cent.); catechu (brown and yellow, about 30 per cent.); sumac or sumach (10 to 18 per cent.); oak bark (6 to 10 per cent.); fir bark (8 to 10 per cent.), and the tannins obtained from these sources (up to 100 per cent.). Tannin is often adulterated by magnesium sulphate. *Prepared catechu* contains additions of alum and potassium bichromate, ammonia, etc.

Each of the above tannins has its own peculiar properties, the tannins or tannic acids of various sources are in no way identical: thus the tannic acid of gall-nuts is a totally different substance from the tannic acid of catechu, etc.

H. R. Procter divides the known tannic acids into 3 groups according to the qualitative reactions they give (*J. S. C. I.*, 1894, 487). Two reagents are utilised: a 1 per cent. solution of iron ammonium alum (A), and bromine water (B).

Group I. Tannins which are precipitated by B and give a greenish-black coloration with A—catechu tannins.

Group II. Tannins which are precipitated by B and give bluish to violet-black colorations with A—mixtures or tannins of undefined character.

Group III. Tannins which are not precipitated by B and give a blue coloration with A—derivatives of pyrogallic acid.

Distinctive Reactions.—Tannin solution + copper sulphate solution + ammonia: a precipitate is always produced, which may dissolve in excess of the precipitant. If it remains undissolved, the tannin belongs to the group of the gall-nut tannic acids, or it contains protocatechuic acid.

Dilute tannin solution + 2 or 3 crystals of sodium nitrite + 3 or 4 drops of very dilute sulphuric acid: a red coloration is generally produced, which slowly turns to purple and dark blue or to green and olive. Frequently a yellow or brown coloration or precipitate is at once produced; then a tannin producing ellagic acid is present (ellagic acid itself and gall-nut tannic acid do not give the reaction).

The tannins of the coniferæ, mimosæ and certain other plants give with a strong hydrochloric acid solution of stannous chloride, 10 c.c. to 1 c.c. of tannin decoction, a pale red precipitate, which appears after about ten minutes.

If, when a tannin decoction is poured on pine splinters, which are moistened before and after drying with strong hydrochloric acid, a pale red or violet coloration is produced, catechu-tannic acid and phloroglucinol are indicated.

A few drops of a decoction of *Knoppern* (galls formed on the acorn cups of a Hungarian species of oak), mixed in a dish with sodium sulphite crystals, give a purple-red coloration.

Qualitative Examination.—Tannin must give as clear a solution in water as possible, and also in ether-alcohol (1 : 1). In the latter solvent there remain undissolved: starch, milk sugar, dextrine, sugar, extractive matters, magnesium and sodium sulphates. Tannin is frequently adulterated with dextrine. A good tannin should contain about 90 per cent. of tannic acid and 7 to 8 per cent. of water. The ash should be low (see also F. Günther, *Ber. d. d. Pharm. Ges.*, 1895, v., 297).

Quantitative Estimation of Tannic Acid.—The proportion of tannic acid, which, as already stated, is extremely variable, determines the value, except in certain cases in which the presence of colouring matter is of importance. Many methods have been proposed for its estimation (see also Fresenius, *Quant. Anal.*, ii., 484), which may be divided, according to the principle of the process, into the following:—

I. Hydrometric methods, II. Titration with permanganate, III. Gravimetric hide-powder methods. The last is continually gaining ground over the two first, and when exactly considered is the most thorough, since it determines directly how much tannin (*i.e.*, a substance forming leathery matter with hide powder) is present. The permanganate method is less reliable, since in this case the nature of the individual tannic acid comes into play and causes abnormalities.

I. The hydrometric methods are said to give favourable results. However, for each separate source of tannic acid a distinct table is necessary, so that in the case of abnormal impurities, adulterations, and unknown tannins the method cannot be used and is thus unsuitable for general use. For details of the hydrometric method see *Praktische Anleitung für Gerber zur Untersuchung der Gerbmaterialien*, by Professor v. Schröder of Tharand (to be obtained from the author, price 2 marks).

II. The permanganate method comprises two operations: (*a*) the titration of the total oxidisable matter; (*b*) titration of the solution freed from tannic acid. The difference, $a - b$, gives the tannic acid.

(*a*) A quantity of the solution containing about 0·1 gramme of dry substance is titrated with $\frac{1}{25}$ N permanganate, using indigo-sulphuric acid as indicator (25 c.c. of a 0·1 per cent. solution of indigotin in sulphuric acid), until the liquid has acquired a yellow coloration. The consumption of permanganate by the 25 c.c. of indigo solution, determined by a separate experiment, is deducted.

(*b*) A second portion of the tannin solution is precipitated with gelatine or hide-powder and an aliquot part of the filtrate (see III.) titrated with permanganate and indigo solution as in *b*. The difference between *a* and *b* gives the tannic acid. The values obtained by titration (number of c.c. of permanganate) are generally not calculated to tannic acid, but to the equivalent quantity of oxalic acid. According to Neubauer and Oser, however, 0·063 gramme of oxalic acid corresponds to 0·04159 gramme of gallotannic acid (tannin) or to 0·06235 gramme of bark-tannic acid. Accordingly

Oxalic acid × 0·666 = tannin
Oxalic acid × 0·99 = bark-tannic acid.

It will be noticed that the equivalents of the various tannic acids are so different that great errors might be the result of applying the method to a mixture of various tannic acids. The process is thus unreliable.

III. The gravimetric hide-powder method is to be regarded as giving the only *absolute* estimation. It comprises the following two single determinations: (*a*) Anhydrous extracted matters soluble in water, and (*b*) Extracted matters *minus* tannins, or "non-tannins".

(*a*) About 10 grammes, or a quantity corresponding to 10 grammes, of the tanning material is dissolved to 1,000 c.c., and 100 c.c. of the liquid, filtered through glass-wool, asbestos, etc. (not through paper, which absorbs tannin), is evaporated and dried at 100° C. to constant weight. The result is the "total extract".

(*b*) A further portion of the filtrate (containing 0·6 to 1·0 gramme of solid matter) is mixed with 10 grammes of the purest hide-powder, added gradually, until tannic acid can no longer be detected in the solution. The mixture is well stirred and when all the tannic acid is absorbed, 50 c.c. are filtered (paper may now be used), evaporated and dried at 100° C. The result gives the "non-tannins".

a – *b* = tannin, which is calculated to 100 parts by weight.

The method is very exact, easily and rapidly carried out and is scientifically unassailable. The purity of the hide-powder is naturally of importance, it ought not to contain any matter soluble in water, for which allowance would otherwise have to be made.

Procter conducts the analysis in the following manner: To 100 c.c. of a tannin solution, not containing more than 0·6 gramme of solids, are added, with intervals of stirring for ten minutes, three quantities of 2 grammes each of well washed and dried hide-powder. The mixture is thoroughly agitated by means of a stirring apparatus working at 320 to 420 revolutions per minute. After thirty minutes the whole of the tannin is absorbed, the liquid is then filtered and 50 c.c. of the filtrate evaporated to dryness. If the result be deducted from the weight of the total extract contained in an equal volume of the decoction, the difference gives the weight of tannin absorbed by the hide-powder. Since Procter does not use quite dry hide-powder, he notes the difference between the originally dry and the washed and pressed hide-powder, by weighing before and after, and allows 1 c.c. of water for each increase in weight of 1 gramme, which in the calculation must be added as a correction to the 100 c.c. of tannin solution. The use of an inconvenient hide-powder filter (see *Der Gerber*, 1887, 137) is not attended by essential advantages and hinders working.

If a complete analysis is required, water, insoluble matter, ash, etc., are determined.

IV. *Technical Examination.*—Tannin analyses should be supplemented by comparative dyeing or weighting trials, which are carried out in a manner dependent on the particular tannin and its proposed use.

In this case, as always, the technical examination must be made as nearly as possible under actual working conditions. For example, 2 grammes of the sample (sumac, mirabolans) are boiled for fifteen minutes with 500 c.c. of water, and the whole decoction, including insoluble matter, made up to 500 c.c. To each beaker are then added 10 grammes of common salt, and at 90° to 95° C. 10 grammes of cotton yarn are suspended in the liquid. After stirring, allowing to cool and standing (in all) three hours, the yarn is taken out, squeezed, and each portion treated with 200 c.c. of basic ferric sulphate of 1·5°B. (1·01 sp.gr.) in a beaker. After stirring for fifteen to twenty minutes the yarn is removed, washed and the depth of the shade compared. Antimony salt may also be used. In a similar manner, comparisons may be made of samples at different prices (see also *Leipziger Färberzeit*, 1896, 261).

Aniline and Aniline Salts.

The value of aniline preparations depends on the proportion of aniline and on neutrality. They must be as dry as possible, free from excess of acid or base and also from other organic compounds.

Aniline Oil, Aniline.—(*a*) Distillation test: 100 c.c. of the sample are distilled; at least 96 per cent. should come over between 180° and 184° C. The distillation must be conducted in a place free from draughts and over a naked flame. The contents of the flask must be in lively ebullition, but must not be overheated. If the aniline is bought to a type sample, the latter should also be distilled in a similar manner at the same time, in order to eliminate differences due to atmospheric variations.

(*b*) Titration with bromine solution in a similar manner to aniline salts (see below).

Aniline Salt (hydrochloride, tartrate, etc.).

Aniline.—(*a*) Titration with bromine solution (Welter-Vaubel). The method depends upon the quantitative conversion of aniline in strong acid solutions into tribromaniline by the action of a solution of bromine in caustic potash (potassium bromide + bromate) and the recognition of excess of bromine by potassium iodide starch paper.

The brominating solution is obtained by gradually adding 100 grammes of commercial bromine (94 to 96 per cent.) to a solution of 95 grammes of caustic potash (73 per cent. of KOH) in 300 to 400 c.c. of water, boiling gently for one hour and filling up to 2,000 c.c. This solution is standardised against chemically pure aniline hydrochloride, dried over sulphuric acid to constant weight: 1 gramme of the pure aniline salt is dissolved to 1,000 c.c. and 100 c.c. of the solution (= 0·1 gramme of substance) is titrated with the above solution, after adding 25 to 50 c.c. of strong hydrobromic acid, until the solution just becomes yellow. Instead of pure hydrobromic acid, hydrochloric acid, to which 10 per cent. of hydrobromic acid has been added, may be used; the same yellow coloration is obtained. Pure hydrochloric acid may be also used, but potassium iodide starch paper must then be employed. For example, if 96 c.c. of bromine solution correspond to 0·1 gramme of chemically pure aniline salt, the percentage of aniline salt in the sample next titrated is given by a simple calculation. Thus if 10 grammes of the sample are dissolved to 1,000 c.c., then 100 c.c. diluted to 1,000 c.c. and 100 c.c. of the last solution require 90 c.c. of bromine solution, then $96 : 0{\cdot}1 = 90 : x$, or $x = 0{\cdot}09375$, corresponding to 93·75 per cent. of aniline hydrochloride.

The method is very accurate, giving results agreeing to 0·1 per cent.

(*b*) The aniline may also be roughly determined in the following manner (Nölting, *Anilinschwarz*): In a stoppered graduated cylinder, 20 grammes of aniline salt, dissolved in 40 c.c. of hot water, are well shaken with 7 grammes of caustic soda (dissolved in 20 c.c. of water) and 30 grammes of common salt. After cooling, the volume is made up with water to 200 c.c. and the volume of the separated oil read off. The percentage by weight of aniline is obtained by multiplying by 5·13.

(*c*) 200 to 300 grammes of aniline salt are decomposed with caustic soda, the oil separated, dried by means of calcium chloride and distilled as given under *Aniline Oil* (see above).

Estimation of Acid.—(*a*) The acid may be determined by ordinary analytical methods. For example, in aniline hydrochloride, the hydrochloric acid can be precipitated by silver nitrate in nitric acid solution; in aniline tartrate, the tartaric acid can be estimated as in tartar, after separation of the aniline oil (see *Tartar*). The excess or deficiency of the acid is then calculated from the percentage of aniline oil.

(*b*) Knecht and Rawson proceed as follows: in each of three flasks, of 200 c.c. capacity, are placed 100 c.c. of water and 1 c.c. of a 0·1 per cent. solution of Crystal Violet; to two of these flasks (No. 1 and No. 2) are added 50 grammes of the sample of aniline salt, the third flask serves for comparison, as also No. 2. Flask No. 1 is then titrated with $\frac{1}{10}$ N caustic soda until the solution, at first blue, again becomes a violet similar to No. 3.

(*c*) The aniline salt solution may also be directly titrated with standard caustic soda and litmus tincture to incipient blue coloration. Phenolphthaleïn is not as suitable.

Free Hydrochloric Acid. Excess of free hydrochloric acid in aniline hydrochloride is recognised by the reaction given by filter paper saturated with a 0·1 per cent. solution of magenta or methyl violet; magenta paper is decolorised by excess of acid, whilst methyl violet is turned green.

Free Aniline.—Free aniline is detected by means of neutral copper sulphate, which imparts a greenish-brown coloration, whilst aniline salt remains colourless. It may also be detected and estimated by Congo Red. This method depends on the property of Congo Red, which has been turned blue by a trace of acid, of being again turned to red by free aniline. In the estimation, titration with $\frac{1}{10}$ N acid is accordingly continued until the blue coloration begins to appear.

Water may be estimated by drying several grammes over sulphuric acid to constant weight.

For the estimation of toluidine in aniline and *vice versâ* see *Zeits. anal. Chem.*, 1895, xxxiv., 734.

Application.—On an extensive scale in the production of aniline black (direct black, oxidation black, steam black, etc.) in dyeing and printing. "Aniline for blue" is pure aniline, "aniline for red" is a mixture of aniline and toluidines.

Thickening and Stiffening Materials.

Starch is generally prepared from potatoes and wheat, less often from maize, rice, barley, etc.

A direct quantitative estimation of the percentage of starch is hardly ever required for technical purposes in the textile industries, since it gives no criterion of the applicability and source of the material. This estimation is therefore not described; details may be found in Fresenius, *Quant. Anal.*, ii., 459, 479 and other analytical text-books. The principle of the process is: the starch is first made into a paste, then brought into solution by means of malt extract (diastase), converted ("inverted") by means of hydrochloric acid into dextrose, and the latter estimated by the reduction of Fehling's solution, gravimetrically (Allihn) or volumetrically (Soxhlet).

Microscopic examination is of the first importance, and quite unavoidable if it is desired to form a comprehensive opinion of the sample. Thanks to the characteristic form and the different dimensions of the grains of the various varieties of starch, it is not difficult, not only to distinguish qualitatively between the different varieties, but also to obtain a certain idea of the purity and uniformity of the material, since inorganic and organic impurities or admixtures, such as sand, mould spores, products of decomposition, etc., etc., may be detected with certainty in this manner. The microscopic appearances of the different starch grains are represented in Plate II. at the end of the book. For further information special works should be consulted: Nägeli, *Die Stärkekörner;* J. König, *Chemie der menschlichen Nahrungs- und Genussmittel;* J. Möller, *Mikroskopie der Nahrungs- und Genussmittel aus dem Pflanzenreiche.* The diagrams in Plate II. are taken from the last-named work.

Water.—Of great importance is also the amount of water, which increases considerably when the starch is stored and may reach 35 per cent. The permissible quantity of water is 20 per cent. in potato starch and 16 per cent. in wheat starch, whilst the normal amounts are 16 to 18 and 14 to 16 per cent. respectively.

(*a*) In order to estimate the water, 10 grammes of starch are first dried for one hour at 40° to 50° C., then for four to five hours at 120° C., and weighed after cooling in the exsiccator. The loss in weight gives the proportion of water.

(*b*) For works where the determination in question is frequently made and several control determinations have to be done in succession, the alcohol method of Scheibler (*Dingl. Polyt. Journ.,* cxcii., 504) is to be recommended, although it makes no claim to absolute accuracy.

The method is based upon the empirical fact that, when 1 part of starch containing 11·4 per cent. of moisture and 2 parts of alcohol of 90 per cent. by volume are mixed, neither takes up water from the other, thus in a sense both are equally hygroscopic; moister starch loses water and dilutes the alcohol; drier starch becomes wetter and the alcohol more concentrated. According to Scheibler, 41·7 grammes of starch are weighed into a glass vessel with an air-tight stopper, 100 c.c. of alcohol of 90° Tr. (90 per cent. by volume) added, the mixture frequently shaken during one hour, filtered through a dry filter and the specific gravity of the filtrate taken. The percentage of water in the starch is then taken direct from the following table :—

Water in starch, per cent.	°Tralles.	Water in starch, per cent.	°Tralles.	Water in starch, per cent.	°Tralles.
0	93·3	14	89·1	28	84·6
1	93·1	15	88·7	29	84·3
2	92·9	16	88·3	30	84·0
3	92·6	17	88·0	31	83·7
4	92·3	18	87·7	32	83·4
5	92·0	19	87·4	33	83·1
6	91·7	20	87·1	34	82·8
7	91·4	21	86·7	35	82·5
8	91·2	22	86·4	36	82·2
9	90·9	23	86·1	37	81·9
10	90·5	24	85·8	38	81·6
11	90·1	25	85·5	39	81·3
12	89·8	26	85·2	40	80·9
13	89·5	27	84·9	50	78·1

Ash.—Important conclusions as to the purity of the sample may occasionally be drawn from an estimation of the ash. The ash in naturally pure starch ranges from 0·5 to 1·0 per cent. If starch is adulterated with sand, gypsum, chalk, barytes, clay, etc., these substances will be found in the ash. The starch may also be brought into solution by means of malt extract, when the above mineral impurities remain undissolved and may be further examined. According to Cailletot, 4 to 5 grammes of finely powdered starch are shaken with chloroform. The specifically lighter starch (sp. gr. 1·4) rises to the surface, whilst most adulterants—which have a greater specific gravity than chloroform (1·526)—sink to the bottom.

Organic impurities may consist, according to Curdes, of coal-dust, dust, residues of potato skin, mould spores, dead algæ, particles of wood, etc. These all remain undissolved when the starch is brought into solution by means of malt extract. A less valuable starch is the only organic adulterant to be considered; it is detected by means of the microscope.

The presence of *gluten* is occasionally important. It is recognised, according to Böttger (*Polyt. Notizbl.*, 1869, No. 15) in the following manner: 1 gramme of starch is heated to boiling with 180 c.c. of water, stirring well with a glass rod. If the starch contains gluten, the foam remains when boiling is stopped; if gluten is absent the foam disappears with the cessation of ebullition. The proportion of gluten may be determined by means of an estimation of nitrogen: pure starch is free from nitrogen, which element is contained in gluten.

Technical Examination.—In addition to the above preliminary tests, practical tests are generally made, according to the purpose for which the starch is required. In this respect are to be considered: the thickening or stiffening capacity of the starch, its property of turning sour, and its general effect upon dye and fibre.

Thickening and Stiffening Test.—A paste and a thinner solution are made of the starch to be examined and of standard samples; comparative trials of thickening or stiffening are then made by means of dressing and padding.

J. Wiesner (Wagner's *Jahresberichte der chem. Tech.*, 1868, 460) conducts the trials as follows: For each sample of starch twenty to thirty threads of cotton of medium size, about 50 cms. long (of finer yarn somewhat shorter, of coarser somewhat longer threads must be taken) are accurately weighed together, each separate thread is then drawn through the starch paste, *uniformly* stripped between the fingers and hung up to dry in an exactly vertical position at the room temperature. The second series of twenty to thirty threads is treated in exactly the same manner with the standard starch, similarly a second, third, etc., sample. The threads, which it is most important not to crease or bend either before or after drying, are then fixed, one at a time, in a clamp in such a manner that at first only a short length of the thread projects upwards above the clamp. The thread is then raised by degrees, being clamped after each rise, until the thread finally falls over by its own weight to one side, so that the extremity lies in the same horizontal plane with the point of support in the clamp. Creased or broken threads are rejected. The length of each thread is then accurately measured from the extremity to the point at which it was clamped and the average length calculated. The threads are then accurately weighed and thus the weight of adherent starch obtained. The stiffening capacity is inversely proportional to the weight of starch and directly proportional to the length of the thread.

Example :—

Starch.	Average length.	Average weight of starch on the thread.
a	l	w
b	l_1	w_1
c	l_2	w_2
d	l_3	w_3

The stiffening capacities are then in the following ratios: $a : b = \frac{l\,w_1}{l_1\,w}$; $a : c = \frac{l\,w_2}{l_2\,w}$, etc., thus if a be taken as unity, $b = \frac{l_1\,w}{l\,w_1}$, $c = \frac{l_2\,w}{l\,w_2}$, $d = \frac{l_3\,w}{l\,w_3}$.

The method necessitates very tedious labour and a careful choice of uniform threads, if reliable results are to be obtained.

Keeping Capacity.—50 grammes of starch are boiled with 1 litre of water, the volume made up to 1 litre, and the solution allowed to stand for several days. That starch, which remains fresh for the longest time, is *ceteris paribus* the best, that which first turns sour and ferments, or exhibits other signs of bacterial decomposition, is the least valuable.

Dressing Test.—Pure white material (for wheat starch) or fabrics dyed in sensitive colours (benzopurpurin, Turkey red, logwood black) are treated with the starch in question on the padding machine, dried and examined. Attention is to be paid to the general appearance, "feel," the stiffening produced, the action on the dye, etc.; comparative trials may also be required.

Application.—In printing, especially calico-printing, also on an equally extensive scale in finishing.

Prepared and Soluble Starch.—A large number of preparations of starch come into commerce under various names, partly as substitutes for gum and size, partly for starch. The majority consist of ordinary starch converted into a soluble form by mechanical or chemical treatment. They are also known as "soluble starch," "vegetable size," etc.

These substances, which differ greatly, are generally prepared by treating starch with superheated steam under pressure, with acids (sulphuric, hydrochloric), with caustic soda, with chlorine, etc. Thus the resulting products may contain the most varied admixtures and impurities, which may be very harmful to the fabrics on which the starch is used. On this account such preparations should never be employed without previous examination. The consumer should convince himself of the harmlessness of the substance by analysis (acids, caustic soda, chlorine) or by a number of carefully performed practical tests. Of a vast number of these preparations only a few may be briefly mentioned.

Soluble starch, obtained by heating starch with water in autoclaves for thirty to sixty minutes, at a pressure of 2 to 3 atmospheres, is a more or less clear gummy mass, which has to a great extent lost the thickening properties of starch.

Apparatin is a clear gummy mass, soluble in water, obtained by boiling starch with caustic soda and then neutralising the excess of soda with sulphuric acid. 60 kilos. of potato starch + 200 litres of water + 12 kilos. of caustic soda of 36° B. (diluted with 60 litres of water) are boiled together. The product is recommended especially for cotton and mixtures of cotton and wool.

Chloïn generally contains free chlorine; it is obtained by the action of bleaching powder on starch. 150 parts of starch are treated with 100 parts of bleaching powder solution of 5° B. and 1,750 parts of water at 60° to 70° C., or at the boiling point for ten minutes. The commercial preparation should be rejected, but it is said to be most suitable for felt, woollens and woollen mixtures.

Starch powder also comes into the market in a soluble form. This is generally a preparation obtained by the action of sulphuric acid under pressure at the boiling point; it is sold both in the form of paste and powder.

Dextrine is a substance soluble in water, obtained by roasting insoluble starch; it comes into the market also under the names of "burnt starch," "British gum," "artificial gum," etc.

Good dextrine should not be hygroscopic, but should be dry, odourless, with a slight insipid taste (not sweet: maltose), readily friable, soluble in an equal volume of water, insoluble in alcohol and of 1·5 specific gravity. It must give with water a solution as colourless, clear and neutral as possible, which is not coloured blue by iodine solution (starch), not rendered turbid by lime water (oxalic acid), not precipitated by tannic acid and baryta water (soluble starch), or by lead acetate (gum Arabic, vegetable mucilage) and hardly reducing Fehling's solution (maltose). Quantitative estimations may be made of: maltose, by reduction of Fehling's solution; starch, as organic matter insoluble in water; sand

and mineral constituents, as ash (the ash of pure dextrine should not appreciably exceed 0·5 per cent); excess of water, by drying 20 to 30 grammes at 110° to 115° C. (the water should not exceed 8 per cent. at the most); and the acidity, by titration with $\frac{1}{10}$ N caustic soda. A microscopic examination must also be undertaken; in the case of dark samples, however, information often cannot be obtained in this way. The keeping capacity may also be tested, as with starch, and the proper practical tests, which indicate the applicability of the material for various requirements, should be carried out (see also Hanofsky, *Mitth. d. k. k. techn. Gewerbemuseums in Wien*, 1889, 56; *J. S. C. I.*, 1889, 573; Böckmann, *Chem.-tech. Unters.*, i., 557; Kellner, *Zündwaarenfabrikation*, p. 51).

Application.—In printing and finishing.

Gum Arabic should consist of irregular shining and brittle lumps of a white to wine-yellow to brown colour, generally traversed by cracks in the interior. It must be readily powdered and not at all hygroscopic, show a shining conchoidal fracture and give with water an almost clear, thick viscous solution, which produces threads. The solution should not be slimy or gelatinous, it may have only a slight acid reaction and must be very adhesive. The taste must be slight and mucilaginous.

Gum Arabic is frequently adulterated by insoluble cherry resin, dextrine, etc., and bleached with sulphurous acid. In the latter case sulphuric acid can be detected. It is also often adulterated with the less valuable Senegal gum, or the latter is sold directly as gum Arabic (Böckmann, Kellner, *loc. cit.*).

For printing purposes it must be quite free from sand and the solution must not turn sour too soon. The keeping capacity should be tested as with starch. 100 grammes of gum dissolved in 1 litre of water should give a solution of about 5° B. The ash should reach only fractions of 1 per cent.

Application.—In printing, especially in the Mulhouse cotton industry on a great scale; also in finishing; in printing on silk.

Gum Senegal, as compared with gum Arabic, forms larger and more transparent spherical lumps, more rarely exhibits cracks, which however then reach to the interior, and frequently has in the interior large tear-shaped air bubbles. Externally it is rougher and less shining, white to reddish yellow in colour; it shows a large conchoidal fracture of high lustre (after Böckmann, Kellner, *loc. cit.*).

According to Liebermann (*Chem. Zeit.*, 1890, 665; *J. S. C. I.*, 1890, 770; 1893, 66) gum Senegal forms lumps of dull appearance, somewhat like etched glass, which, however, are shining and transparent in the interior. The lumps are generally longish, straight or twisted, cylindrical, annulated like a worm, and thus somewhat mulberry-shaped. Gum Senegal can accordingly be distinguished from gum Arabic by external appearance alone.

Further it differs from gum Arabic in giving only a slight turbidity with mercurous nitrate and in being considerably thickened by borax; it is soluble in water with more difficulty, more slimy and gelatinous — thus of less binding power—and is more readily curdled by many reagents. The last property explains the smaller worth of gum Senegal as compared with gum Arabic.

Application.—As a thickener similarly to gum Arabic.

Gum tragacanth comes into the market in many varieties. It should be without smell and taste, transparent, horny and tough, so that it is very difficult to powder. Only a small portion dissolves in water, the greater part swells up to mucilage, which is not sticky though adhesive (Böckmann, *loc. cit.*).

It ought not to turn sour too rapidly and should contain no foreign constituents (ash).

A technical experiment must be employed in order to evaluate tragacanth ; the stiffening property is the most important (see stiffening property under *Starch*).

Application.—In combination with potato and especially wheat starch (with which it works better), size, dextrine, etc., it is much used in finishing silk (France) and silk mixtures, also for other fabrics. It is also used by printers, who often boil the tragacanth six to ten hours in order to obtain complete division through the water.

A cheap substitute for tragacanth is proposed by Boschan : 20 parts of starch, 6 parts of size and 2 parts of glycerin are violently boiled together in water.

Glue, Size.—A distinction is made between hide, bone and fish glue. Gelatine is purified and bleached bone glue, it is examined in a similar manner to the crude glues, with the addition that regard must be paid to the colour and to substances which might be introduced in bleaching (sulphuric acid by sulphur dioxide, etc.). Hide glue has greater adhesive power than bone glue.

In the first, glue must be free from acid (hydrochloric, etc.) and from the alum used in clearing it. Good glue is also not too dark in colour, is broken with difficulty, has a certain elasticity and is not hygroscopic. The fracture must shine like glass ; a splintery fracture indicates incompletely dissolved sinewy parts. Good glue should not alter in shape in cold water ; it should only swell largely and not even be liquid after forty-eight hours (good hide glue, however, liquefies to some extent). At 48° C. the swollen glue begins to melt and is fluid at 50° C. The adhesiveness of a glue is greater the less its solution has been heated and the better the materials employed.

Glue should have no salt or acid taste. If an accurately weighed quantity of glue be left in cold water for twenty-four hours, and then again dried, that glue is the best which approaches most nearly to the original weight; the reverse is also true (after Böckmann and Kellner, *loc. cit.*).

The following estimations may also be made :—

Water.—Two to three grammes of finely rasped glue are dried at 110° to 115° C. to constant weight.

Ash.—The ash decides between hide and bone glue. The ash of the latter melts in the Bunsen flame, its aqueous solution is neutral, it contains phosphoric acid and chlorine. The ash of hide or leather glue is infusible in the Bunsen flame, contains much caustic lime, is strongly alkaline and generally free from phosphoric acid and chlorine.

Acid.—80 c.c. of water are poured over 30 grammes of glue and allowed to stand several hours. The volatile acids are then driven over in steam, caught in a receiver and, when 200 c.c. have distilled, titrated.

The drying capacity, foreign matter, smell, etc., may also be tested. For further information see Böckmann, *Zündwaaren*, p. 533 *et seq.;* C. Stelling, *Chem. Zeit.*, 1897, 47, 461.

Practical Test.—In addition to the above examination, several finishing experiments should be made, as was the case with starch. Silk mixtures and woollen goods are the best for the purpose. A sensitive benzidine (benzopurpurin) dye, aniline black or logwood black is suitable.

Application.—Very extensive in finishing, less in printing and dyeing (brightening silk).

DYES.

To deal with the subject of the coal-tar dyes lies outside the scope of this work. The subject is sufficiently immense to require a separate treatise. Indeed, there exist numerous excellent books, sufficient for any requirement, treating of the dyes from every point of view; in fact it may be said that the literature of the subject is increased by at least one large work almost every year. A number of books are recommended at the conclusion. In this place it is merely necessary to indicate very briefly the manner of examining dyes chemically and for shade.

The most important characters of a dye are: its chemical composition, general character and properties, shade, strength or concentration, purity and presence or absence of other dyes.

The *chemical constitution* is ascertained by advanced synthetic and analytical methods, which require in the first place a scientifically trained chemist.

The *general character* is studied by means of trial dyeings on different fibres, by different methods and with various assistant materials. The dye fixed on the fibre is examined for fastness to light, air, washing, fulling, soap, water, acids, alkalis, dirt, perspiration, rubbing, chlorine, sulphur, ironing, steaming and moisture, also for equal dyeing and possibility of using in conjunction with other dyes, etc.

The *shade* is ascertained by trial dyeings, using different percentages of dye, in comparison with similar and competing dyes in varying depths of hue, in combination with various dyes used for shading the principal dye, sampling by direct and indirect daylight, by gas-light and electric light, etc.

The *strength* or *concentration* is tested by quantitative dye trials against a standard, or occasionally colorimetrically.

The *purity* and *individuality* are ascertained by qualitative and quantitative estimations of the diluent and foreign colouring matter, intentionally or accidentally present. The following substances are most frequently used as diluents: dextrine, sugar, starch, common salt, Glauber's salt, etc. Dyes, diluted by these agents and containing only 3 per cent. of colouring matter, come into the market—chiefly for export.

The added substances may almost always be discovered as an insoluble residue by fractional solution of the dye in alcohol, ether, etc.

It is frequently more difficult to discover the addition of a foreign colouring matter, used to alter the shade. If the mixture has been made by bringing together the already dry colouring matters, the components may be recognised by dusting the very finely ground dye against wet filter paper. But if the mixture has been more thorough, *e.g.*, as obtained by producing mixed crystals of the components, they are only discovered with much more difficulty, and at times only by an expert by means of a series of typical colour reactions.

In conclusion the following books and articles are mentioned, which treat, among other subjects, of the examination of dyes, more or less from different points of view:—

R. Fresenius, "Bestimmung des Arsens in Farbstoffen," *Zeits. anal. Chem.*, 1888, 179 *et seq.; J. S. C. I.*, 1888, 456.

P. Friedländer, *Fortschritte der Theerfabrikation und verwandter Industriezweige*, i., 1877-87; ii., 1887-90; iii., 1891-94.

Ganswindt, *Lehrbuch der Baumwollgarn-Färberei.*

G. v. Georgievics, *Lehrbuch der chemischen Technologie der Gespinnstfasern.*

G. v. Georgievics, *Lehrbuch der Farbenchemie.*

J. Herzfeld, *Das Färben und Bleichen von Baumwolle, Wolle,* etc. (3 Theile).

Hummel-Knecht, *Die Färberei und Bleicherei der Gespinnstfasern.*

Knecht, Rawson and Löwenthal, *A Manual of Dyeing.*

E. Lauber, *Praktisches Handbuch des Zeugdrucks.*

H. Lehne, *Tabellarische Uebersicht über die künstlichen organischen Farbstoffe und ihre Anwendang in Färberei und Zeugdruck.*

R. Mohlau, *Organische Farbstoffe, welche in der Textilindustrie Verwendung finden.*

R. Nietzki, *Chemistry of the Organic Dye-stuffs.*

G. Schultz, *Die Chemie des Steinkohlentheers mit besonderer Berücksichtung der künstlichen organischen Farbstoffe.*

G. Schultz and P. Julius, *Systematic Survey of the Artificial Organic Colouring Matters.*

E. Weingärtner, "Anleitung zur Untersuchung der im Handel vorkommenden künstlichen Farbstoffe," *Chem. Zeit.*, 1887, Nos. 10 and 12.

O. N. Witt, *Chemische Technologie der Gespinnstfasern; ihre Geschichte, Gewinnung, Verarbeitung und Veredelung.*

O. N. Witt, "Versuch einer qualitativen Analyse der im Handel vorkommenden Farbstoffe," *Zeits. anal. Chem.*, 1887, 100 *et seq.*.

Atomic Weights of the Elements.[1]

Name.	Symbol of the Atom.	Valency.	Atomic Weight.		
			International O = 16.	Didactic H = 1.	Commonly used H = 1.
Aluminium	Al	III, IV	27·1	26·9	27
Antimony	Sb	III, V	120	119·1	120
Argon	A	—	39·9	39·6	—
Arsenic	As	III, V	75·0	74·4	75
Barium	Ba	II, IV	137·4	136·4	137
Beryllium	Be	II or III	9·1	9·03	9·4
Bismuth	Bi	III, V	208·5	206·9	208
Boron	B	III, V	11	10·9	11
Bromine	Br	I, III, V, VII	79·96	79·36	79
Cadmium	Cd	II	112·4	111·6	112
Caesium	Cs	I	133	132	133
Calcium	Ca	II	40	39·7	40
Carbon	C	II, IV	12·00	11·91	12
Cerium	Ce	III, IV	140	139	—
Chlorine	Cl	I, III, V, VII	35·45	35·18	35·4
Chromium	Cr	III, IV, VI	52·1	51·7	52·5
Cobalt	Co	II, IV	59·0	58·56	59
Copper	Cu	I, II	63·6	63·1	63
Erbium	Er	II	166	164·8	169 (?)
Fluorine	F	I	19	18·9	19
Gadolinium	Gd	III	156	155	—
Gallium	Ga	IV	70	69·5	69
Germanium	Ge	IV	72	71·5	—
Gold	Au	I, III	197·2	195·7	196·7
Helium	He	—	4	4	—
Hydrogen	H	I	1·008	1·00	1
Indium	In	III	114	113·1	113·4
Iodine	I	I, III, V, VII	126·85	125·9	127
Iron	Fe	II, III. IV, VI	56·0	55·6	56
Iridium	Ir	II, IV, VI	193·0	191·5	192·5
Krypton	Kr	—	81·8	81·2	—
Lanthanum	La	IV	138	137	139
Lead	Pb	II, IV	206·9	205·35	207
Lithium	Li	I	7·03	6·98	7
Magnesium	Mg	II	24·36	24·18	24

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[1] The atomic weights inserted in the columns headed "International" and "Didactic" are those given for 1901 by the Atomic Weight Commission of the German Chemical Society.

Atomic Weights of the Elements.—continued.

Name.	Symbol of the Atom.	Valency.	Atomic Weight.		
			International O = 16.	Didactic H = 1	Commonly used H = 1.
Manganese	Mn	II, IV, VI, VII	55·0	54·6	55
Mercury	Hg	I, II	200·3	198·8	200
Molybdenum	Mo	II, III, IV, VI	96·0	95·3	96
Neodymium	Nd	IV	143·6	142·5	—
Neon	Ne	—	20	19·9	—
Nickel	Ni	II, III, IV	58·7	58·3	58·8
Nitrogen	N	III, V	14·04	13·93	14
Niobium	Nb	V	94	93·3	94
Osmium	Os	II, III, IV, VIII	191	189·6	190
Oxygen	O	II	16·00	15·88	16
Palladium	Pd	II, IV, VI	106	105·2	106·5
Phosphorus	P	III, V	31·0	30·77	31
Platinum	Pt	II, IV, VI	194·8	193·3	197 (?)
Potassium	K	I	39·15	38·86	39
Praseodymium	Pr	IV	140·5	139·4	—
Rhodium	Rh	II, IV, VI	103·0	102·2	104
Rubidium	Rb	I	85·4	84·76	85
Ruthenium	Ru	II, IV, VI, VIII	101·7	100·9	104 (?)
Samarium	Sa	III	150	148·9	—
Scandium	Sc	IV	44·1	43·8	—
Selenium	Se	II, IV, VI	79·1	78·5	79
Silicon	Si	IV	28·4	28·2	28
Silver	Ag	I	107·93	107·12	108
Sodium	Na	I	23·05	22·88	23
Strontium	Sr	II	87·6	86·94	87·5
Sulphur	S	II, IV, VI, VIII	32·06	31·83	32
Tantalum	Ta	V	183	181·6	182
Tellurium	Te	II, IV, VI	127	126	127
Thallium	Tl	I, III	204·1	202·6	204
Thorium	Th	IV	232·5	230·8	231·5
Thulium	Tu	—	171	170	—
Tin	Sn	II, IV	118·5	117·6	118
Tintanium	Ti	IV	48·1	47·7	48
Tungsten	W	IV, VI	184	182·6	184
Uranium	U	IV, VI	239·5	237·7	240
Vanadium	V	III, V	51·2	50·8	51·2
Xenon	X	—	128	127	—
Ytterbium	Yb	III	173	172	—
Yttrium	Y	IV	89	88·3	89
Zinc	Zn	II	65·4	64·9	65
Zirconium	Zr	IV	90·7	90·0	90

Molecular Weights of Certain Compounds (O = 16).

Compound.	Molecular Weight.
Aluminium—	
Al_2O_3	102·2
$Al_2(OH)_6$	156·248
Antimony—	
Sb_2S_3	336·18
Sb_2S_5	400·3
Sb_2O_3	288·
Sb_2O_5	320·
Arsenic—	
$Mg_2As_2O_7$	310·72
$(MgNH_4AsO_4)_2.H_2O$	380·88
As_2S_3	246·18
As_2S_5	310·3
As_2O_3	198·0
As_2O_5	230·0
Barium—	
$BaSO_4$	233·46
BaO	153·4
$BaCO_3$	197·4
$BaCrO_4$	253·5
Bismuth—	
Bi_2O_3	465·0
Bi_2S_3	513·18
$BiOCl$	259·95
$BiAsO_4$	347·5
Boron—	
B_2O_3	70
KBF_4	126·15
Bromine—	
$AgBr$	187·89
HBr	80·968
Br_2O_5	239·92
Cadmium—	
CdS	144·46
CdO	128·4
Calcium—	
CaO	56
$CaSO_4$	136·06
$CaCO_3$	100
Carbon—	
CO_2	44·00
CN	26·04
CO	28·00
HCN	27·048
Chlorine—	
$AgCl$	143·38
HCl	36·458
Cl_2O_5	150·90
Chromium—	
Cr_2O_3	152·2
CrO_3	100·1
$PbCrO_4$	323·0
$BaCrO_4$	253·5
Cobalt—	
CoO	75·0
Co_3O_4	241·0
$K_3Co(NO_2)_6$	452·69
Copper—	
CuO	79·6
Cu_2S	159·26
Fluorine—	
CaF_2	78
KBF_4	126·15
HF	20·008
$BaSiF_6$	279·8
K_2SiF_6	220·7
H_2SiF_6	144·4
Hydrogen—	
H_2O	18·016
Iodine—	
AgI	234·78
HI	127·858
PbI_2	460·6
Iron—	
FeO	72·0
Fe_2O_3	160·0
$Fe_2(PO_4)_2$	302·0
Lead—	
$PbSO_4$	302·96
PbS	238·96
PbO	222·9
$PbCl_2$	277·8
$PbCrO_4$	323·0
Lithium—	
$LiCl$	42·48
Li_2SO_4	110·12
Li_2O	30·06

Molecular Weights of Certain Compounds (O = 16).—cont.

Compound.	Molecular Weight.	Compound.	Molecular Weight.
Lithium—continued—		*Potassium*—continued	
Li_2CO_3	74·06	K_2PtCl_6	485·8
Li_3PO_4	116·09	K_2O	94·30
Magnesium—		K_2SiF_6	220·7
$Mg_2P_2O_7$	222·72	*Silicon*—	
MgO	40·36	SiO_2	60·4
$MgNH_4AsO_4 . \frac{1}{2}H_2O$	190·436	SiF_4	104·4
$Mg_2As_2O_7$	310·72	H_2SiF_6	144·4
$MgSO_4$	120·36	K_2SiF_6	220·7
Manganese—		$BaSiF_6$	279·8
$MnSO_4$	151·06	*Silver*—	
MnS	87·06	Ag_2O	231·86
Mn_3O_4	229·0	$AgCl$	143·38
Mn_2O_3	158·0	$AgCN$	133·97
MnO	71·0	Ag_3PO_4	418·79
$KMnO_4$	158·15	$Ag_4P_2O_7$	605·72
Mercury—		*Sodium*—	
HgS	232·36	$NaCl$	58·50
HgO	216·3	Na_2SO_4	142·16
Hg_2O	416·6	Na_2CO_3	106·10
Hg_2Cl_2	471·5	Na_2O	62·10
Nickel—		*Strontium*—	
NiO	74·7	$SrSO_4$	183·66
$NiSO_4$	154·76	$SrCO_3$	147·6
Nitrogen—		SrO	103·6
N_2O_5	108·08	*Sulphur*—	
N_2O_3	76·08	As_2S_3	246·18
NH_4Cl	53·522	H_2S	34·076
$(NH_4)_2SO_4$	132·204	SO_2	64·06
Phosphorus—		SO_3	80·06
$Mg_2P_2O_7$.	222·72	H_2SO_4	98·076
$Ag_4P_2O_7$	605·72	*Tin*—	
$Fe_2(PO_4)_2$	302·0	SnO_2	150·5
P_2O_5	142·0	SnO	134·5
Platinum—		*Uranium*—	
K_2PtCl_6	485·8	$U_2P_2O_{11}$	723·0
$(NH_4)_2PtCl_6$	443·644	UO_2	271·5
Potassium—		*Zinc*—	
KCl	74·60	ZnS	97·4
K_2SO_4	174·36	ZnO	81·4

Gravimetric Equivalents.

Obtained.	Factor.	Sought.
AgCl	× 0·2474	= Cl
,,	× 0·2543	= HCl
,,	× 0·7526	= Ag
Al_2O_3	× 0·534	= Al
As_2S_3	× 0·6097	= As
,,	× 0·805	= As_2O_3
,,	× 0·935	= As_2O_5
$BaSO_4$	× 0·6566	= BaO
,,	× 0·588	= Ba
,,	× 0·3433	= SO_3
,,	× 0·137	= S
,,	× 0·2747	= SO_2
CO_2	× 0·2727	= C
$CaCO_3$	× 0·44	= CO_2
CaO	× 0·7143	= Ca
$CaSO_4$	× 0·4117	= CaO
Cr_2O_3	× 1·3158	= CrO_3
CuO	× 0·7987	= Cu
,,	× 1·306	= Tannin
Cu_2S	× 0·7987	= Cu
Fe_2O_3	× 0·7	= Fe
,,	× 0·9	= FeO
H_2O	× 0·1111	= H
KCl	× 0·6309	= K_2O
,,	× 0·5235	= K
K_2PtCl_6	× 0·307	= KCl
,,	× 0·159	= K

Obtained.	Factor.	Sought.
K_2PtCl_6	× 0·193	= K_2O
K_2SO_4	× 0·5402	= K_2O
$MgNH_4AsO_4$	× 0·547	= As_2O_3
,,	× 0·636	= As_2O_5
MgO	× 0·6	= Mg
$Mg_2P_2O_7$	× 0·3604	= MgO
,,	× 0·6396	= P_2O_5
,,	× 0·757	= $MgCO_3$
N	× 3·857	= HNO_3
,,	× 1·214	= NH_3
,,	× 3·857	= N_2O_5
,,	× 6·25	= Proteïns
Na_2CO_3	× 0·585	= Na_2O
NaCl	× 0·53	= Na_2O
,,	× 0·3931	= Na
,,	× 0·6068	= Cl
Na_2SO_4	× 0·4366	= Na_2O
$PbCrO_4$	× 0·3096	= CrO_3
PbO	× 0·9282	= Pb
$PbSO_4$	× 0·736	= PbO
,,	× 0·6832	= Pb
Sb_2O_3	× 0·8365	= Sb
Sb_2S_3	× 0·7176	= Sb
SnO_2	× 0·7867	= Sn
ZnO	× 0·8025	= Zn
ZnS	× 0·835	= ZnO
,,	× 0·6701	= Zn

Volumetric Equivalents.

1 c.c. of	N acid	. . . . . = 0·031	grm.	Na_2O.
,,	,,	. . . . . = 0·04	,,	NaOH.
,,	,,	. . . . . = 0·047	,,	K_2O.
,,	,,	. . . . . = 0·056	,,	KOH.
,,	,,	. . . . . = 0·017	,,	NH_3.
,,	,,	. . . . . = 0·053	,,	Na_2CO_3.
,,	,,	. . . . . = 0·143	,,	Na_2CO_3. $10H_2O$.
,,	,,	. . . . . = 0·069	,,	K_2CO_3.
,,	,,	. . . . . = 0·1575	,,	$Ba(OH)_2$. $2H_2O$.
,,	,,	. . . . . = 0·0765	,,	BaO.
,,	,,	. . . . . = 0·05	,,	$CaCO_3$.
,,	,,	. . . . . = 0·028	,,	CaO.
,,	,,	. . . . . = 0·022	,,	CO_2.
,,	,,	. . . . . = 0·191	,,	$Na_2B_4O_7$. $10H_2O$.

1 c.c. of N alkali	= 0·04	grm.	SO_3.
,, ,,	= 0·049	,,	H_2SO_4.
,, ,,	= 0·0364	,,	HCl.
,, ,,	= 0·063	,,	HNO_3.
,, ,,	= 0·054	,,	N_2O_5.
,, ,,	= 0·06	,,	acetic acid.
,, ,,	= 0·063	,,	crys. oxalic acid.
,, ,,	= 0·09	,,	lactic acid.
,, ,,	= 0·075	,,	tartaric acid.
,, ,,	= 0·188	,,	tartar.
,, ,,	= 0·07	,,	crys. citric acid.
1 c.c. of N oxalic acid	= 0·0316	,,	$KMnO_4$.
1 c.c. of $\frac{1}{10}$ N sulphocyanide	= 0·012475	,,	$CuSO_4 . 5H_2O$.
1 c.c. of $\frac{1}{10}$ N thiosulphate	= 0·00354	,,	active Cl.
1 c.c. of $\frac{1}{10}$ N permanganate	= 0·0063	,,	crys. oxalic acid.
,, ,,	= 0·0056	,,	Fe.
,, ,,	= 0·0072	,,	FeO.
,, ,,	= 0·008	,,	Fe_2O_3.
,, ,,	= 0·0278	,,	$FeSO_4 . 7H_2O$.
,, ,,	= 0·0392	,,	$FeSO_4 . (NH_4)_2SO_4 . 6H_2O$.
,, ,,	= 0·01125	,,	$SnCl_2, 2H_2O$.
,, ,,	= 0·0017	,,	H_2O_2.
,, ,,	= 0·00845	,,	BaO_2.
,, ,,	= 0·00345	,,	$NaNO_2$.
,, ,,	= 0·0422	,,	$K_4Fe(CN)_6 . 3H_2O$.
1 c.c. of $\frac{1}{10}$ N iodine	= 0·0032	,,	SO_2.
,, ,,	= 0·00495	,,	As_2O_3.
,, ,,	= 0·00575	,,	As_2O_5.
,, ,,	= 0·01125	,,	$SnCl_2 . 2H_2O$.
,, ,,	= 0·0094	,,	$SnCl_2$.
,, ,,	= 0·0059	,,	Sn.
,, ,,	= 0·0158	,,	$Na_2S_2O_3$.
1 c.c. of $\frac{1}{10}$ N silver nitrate	= 0·00354	,,	Cl.
,, ,,	= 0·00364	,,	HCl.
,, ,,	= 0·00584	,,	$NaCl$.
1 c.c. of $\frac{1}{10}$ N sodium arsenite	= 0·00354	,,	active Cl.
,, ,,	= 0·006	,,	Sb.
,, ,,	= 0·0072	,,	Sb_2O_3.
1 c.c. of $\frac{1}{10}$ N potassium bichromate	= 0·0072	,,	Fe.
,, ,,	= 0·0278	,,	$FeSO_4 . 7H_2O$.
1 grm. of $FeSO_4 . (NH_4)_2SO_4 . 6H_2O$	= 0·1253	,,	$K_2Cr_2O_7$.
,, ,,	= 0·0853	,,	CrO_3.
1 grm. of potassium bichromate	= 0·662	,,	metallic zinc.

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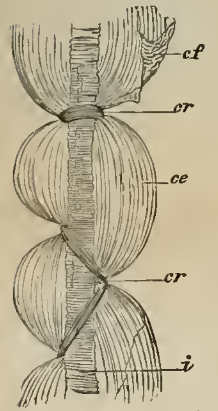
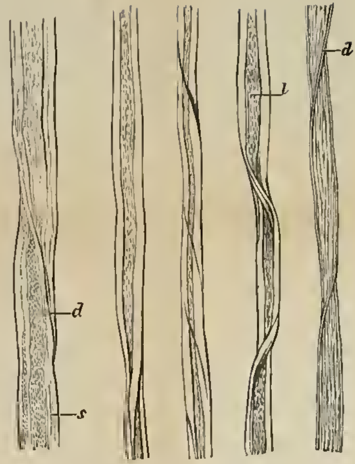
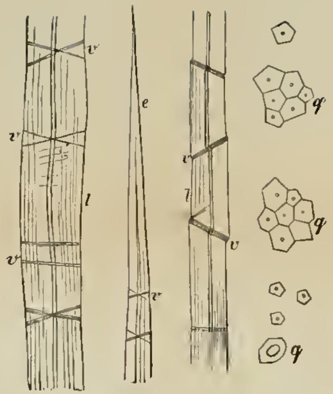


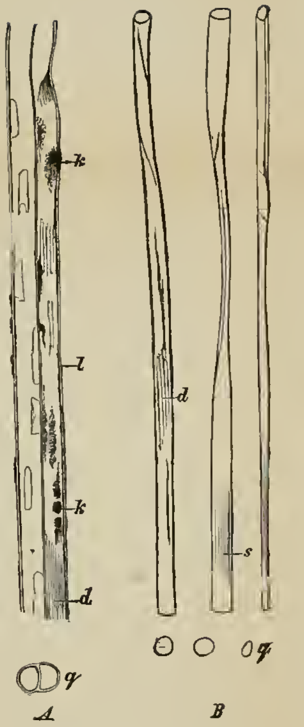
Cotton Swollen in Ammoniacal Copper-Oxide Solution.
cf, fragments of cuticle; *cr*, ring of cuticle; *ce*, protuberent cellulose; *i*, the protoplasmic interior.
(After v. Höhnel.)



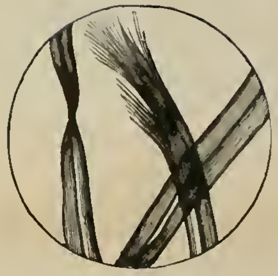
Cotton Fibres (after v. Höhnel).
l, lumen; *d*, position of a twist; *s*, roughnesses of the surface of the cuticle.



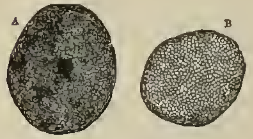
Linen Fibre (after v. Höhnel).
l, longitudinal view; *v*, joints; *q*, sections; *e*, end.



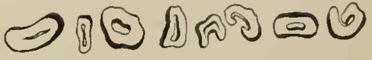
Silk (after v. Höhnel).
A, unboiled; *B*, boiled; *k*, granular lumps upon the sericine layer *l*; *d*, fibroine thread; *q*, sections.



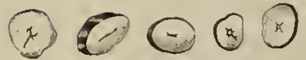
Microscopic Appearance of Tussur Silk.



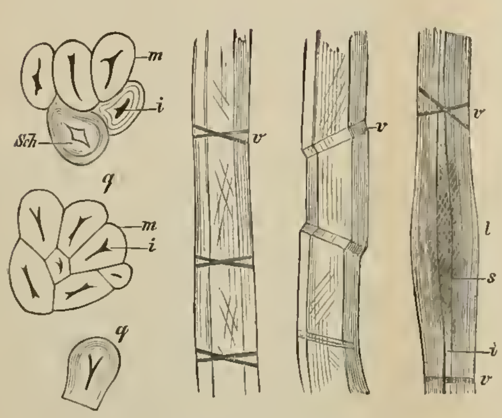
Sections of Typical Wool Fibres.



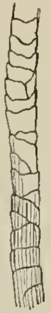
Sections of Cotton Fibres.



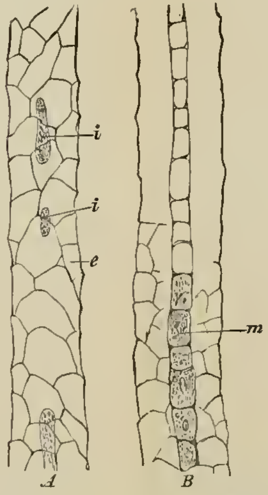
Sections of Cotton Fibres after treatment with Caustic Soda.



Hemp Fibre (after v. Höhnel).
q, sections with central lamellæ *m*, surface layer *Sch*, lumina *i*; *v*, joints; *s*, markings.

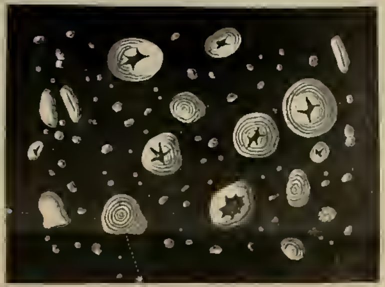


Finest Wool Fibre.

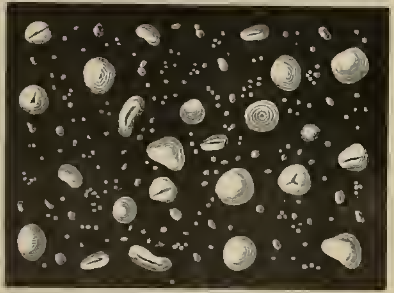


English Leicester Wool (after v. Hohnel).
A, fibre with disconnected medulla *i*; *B*, fibre with cylinder of medulla *m*.

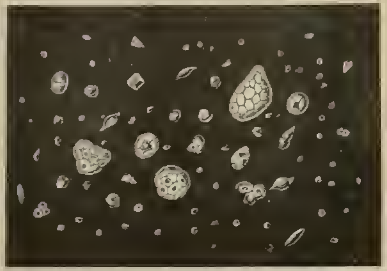
PLATE II.
(*See page* 128.)



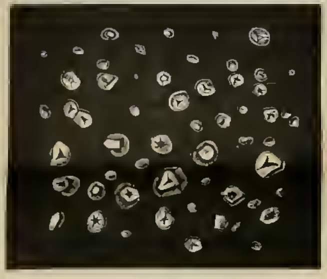
Rye Starch (× 300).



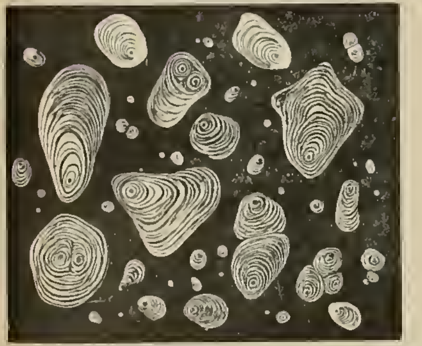
Barley Starch (× 300).



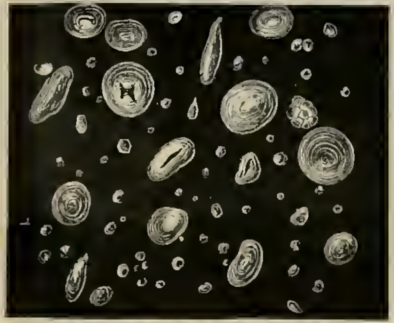
Oat Starch (× 300).



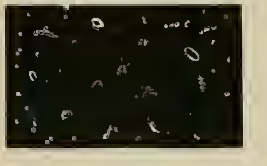
Maize Starch (× 300).



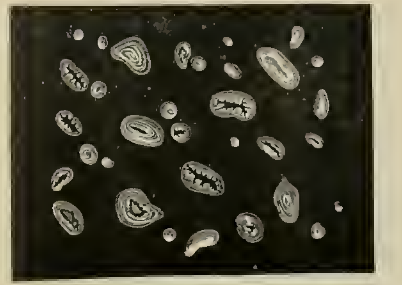
Potato Starch (× 300).



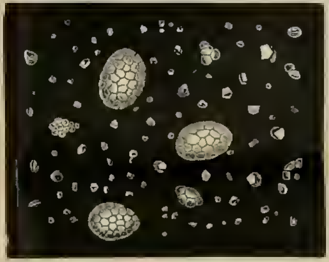
Wheat Starch (× 300).



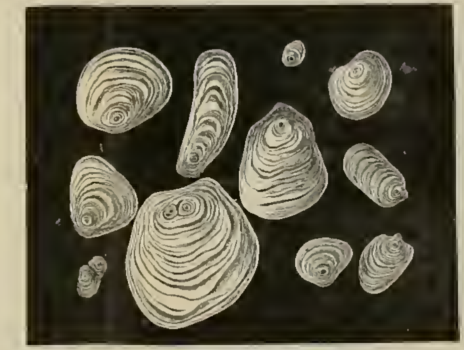
Buckwheat Starch (× 300).



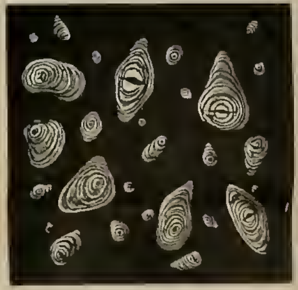
Lentil Starch (× 300).



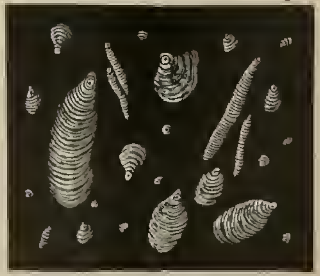
Rice Starch (× 300).



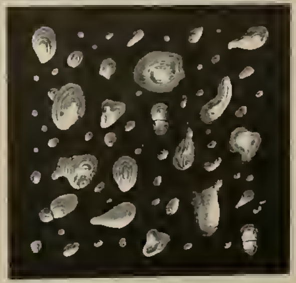
Canna Starch (× 300).



Arrowroot (*maranta*) Starch (× 300).



Turmeric Starch (× 300).



Horse-chestnut Starch (× 300).

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